Normal Incidence Diffraction Beamline

(Exact Bragg Backscattering Beamline)

Esen Ercan Alp

Advanced Photon Source

Argonne National Laboratory

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@ APS

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Plan of presentation

1. General overview of Normal Incidence Diffraction Beamline (NID) or (Exact Bragg Backscattering, EBB)

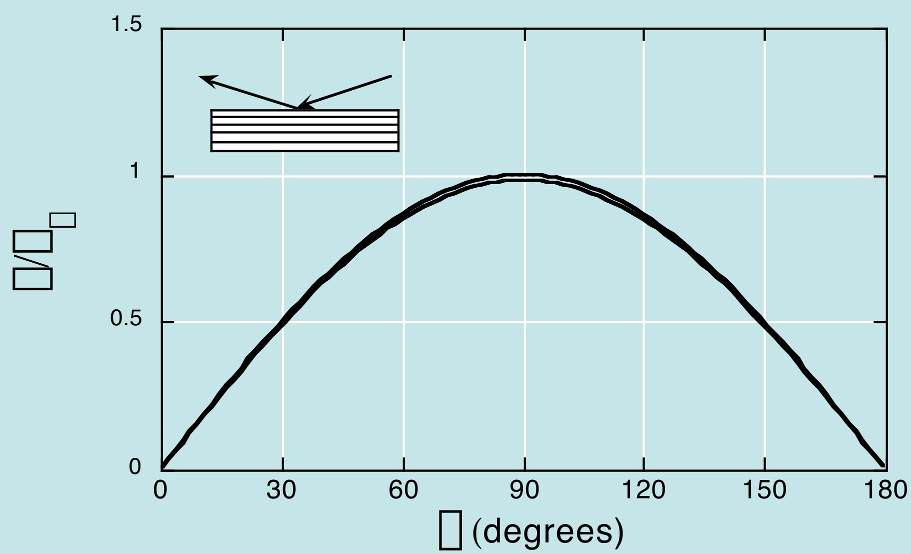
2. Unique aspects and challenges

3. Planned scientific program

   Inelastic x-ray scattering at low energies
   Nuclear resonant scattering at high energies
   X-Ray metrology and high precision measurements
   Coherent imaging

What is Exact Bragg Backscattering ?

First fully treated by Kohra and Matsushita (1972)



$$\frac{\Delta E}{E} = |\chi_H| \quad \sim \mathrm{meV}$$

$$\Delta\theta = |2\chi_H|^{1/2} \sim \mathrm{mrad}$$

$$\chi_H = -r_e \frac{\lambda^2}{\pi V} F_H \sim 10^{-5}$$

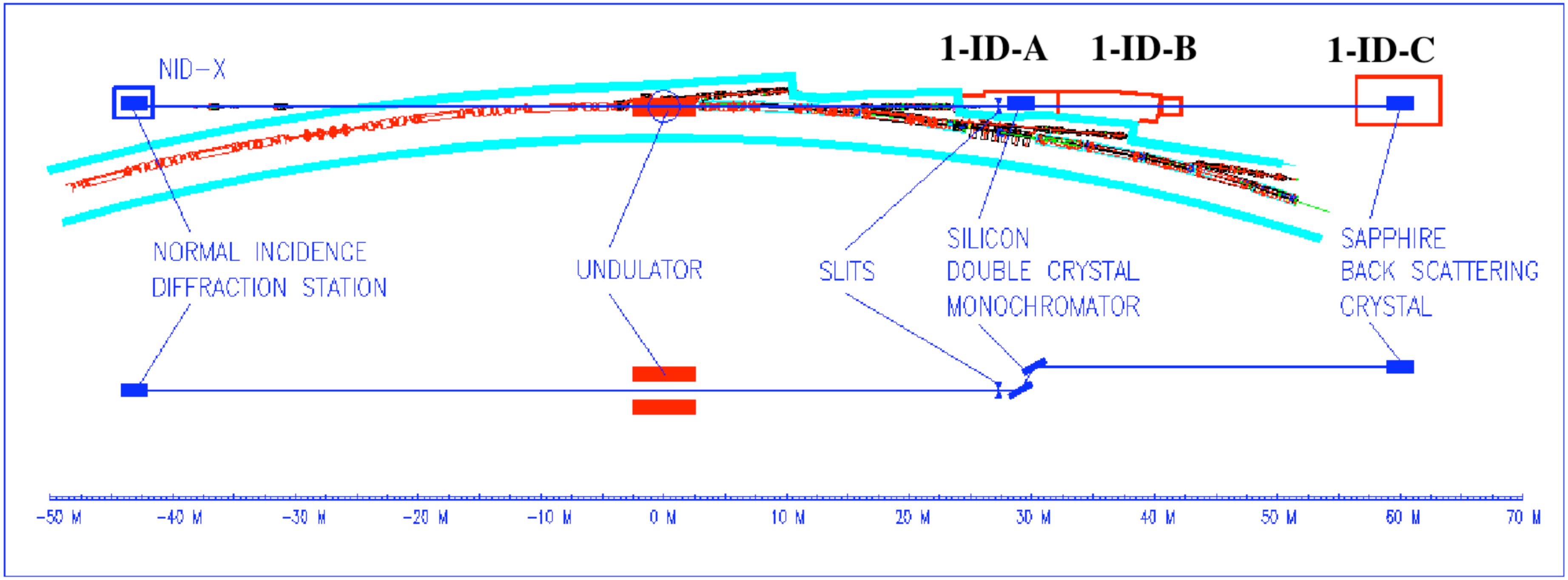
On exact back-scattering, ΔE is the smallest,
and Δθ is the largest of any other Bragg peak.

Early work proposing a back-scattering type of a beamline

1. R. Collela, R. and A. Luccio
   Proposal for Free Electron Laser in the X-ray Region,
   Phys. Letters, 50, (1984) 41.

2. S. Caticha-Ellis, R. Boyce, Herman Winick,
   Nucl. Instr. Meth. A 291 (1990) 132.

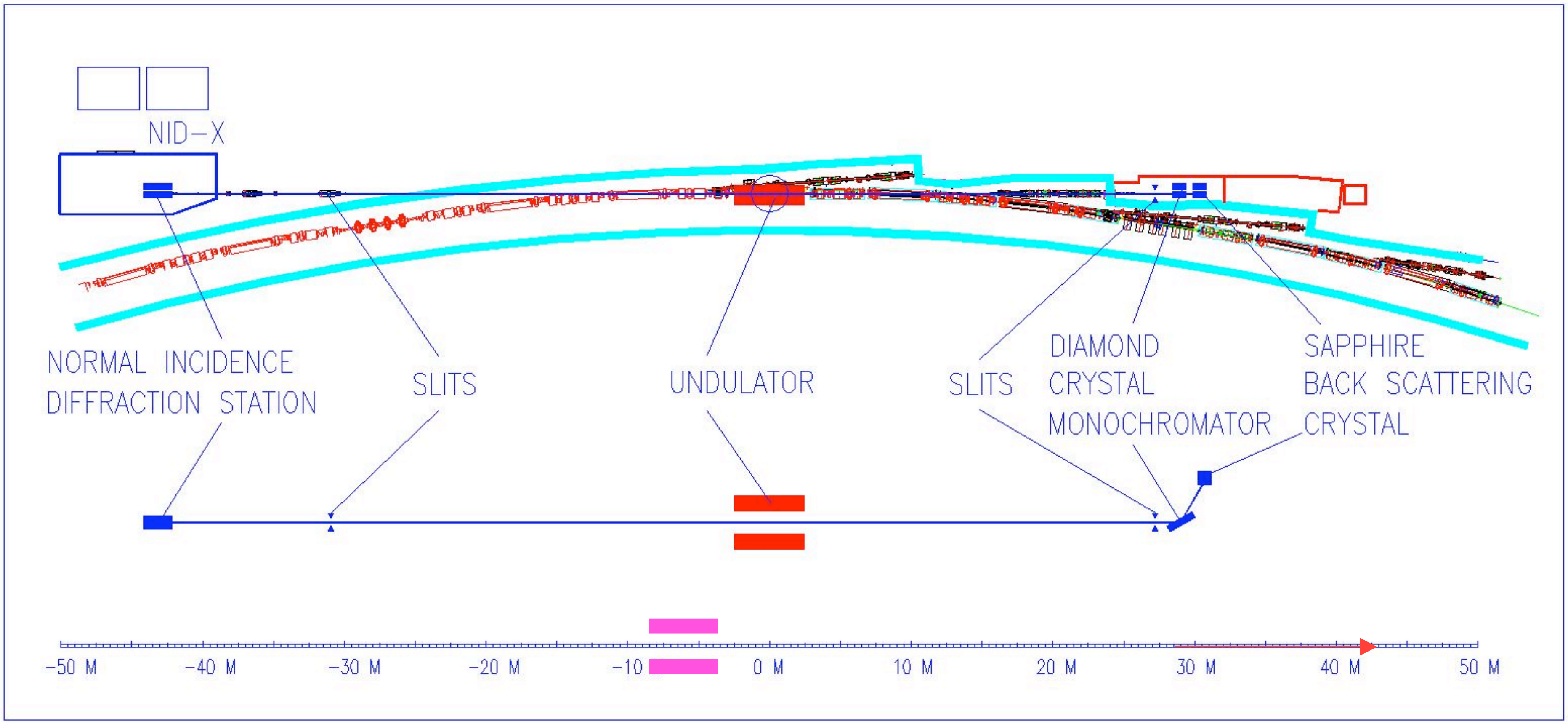
The current layout



The schematic layout of the NID beamline developed for the proof-of-principle experiments. The x-rays produced by the undulator first travels to the right, and they are diffracted back onto themselves by the saphhire crystal, travels back through the Si (111) and the undulator to reach NID-X station.

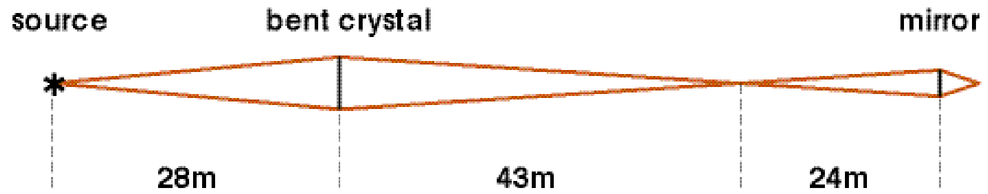
The total distance traveled from the undulator to the NID-X station is 166 m.

The proposed layout



NID-X focusing

horizontal



- beam size at mirror : 1.3mm (4.5mm without crystal)
- focal spot size : <100µm
- mirror length : > E/keV * 0.03m

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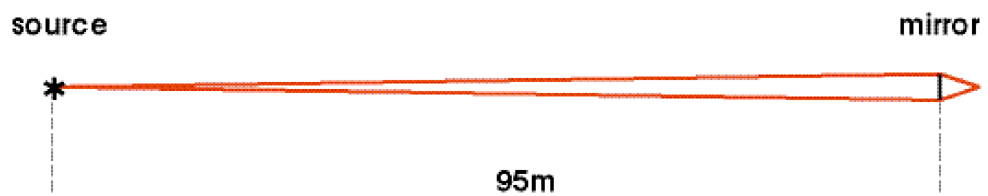
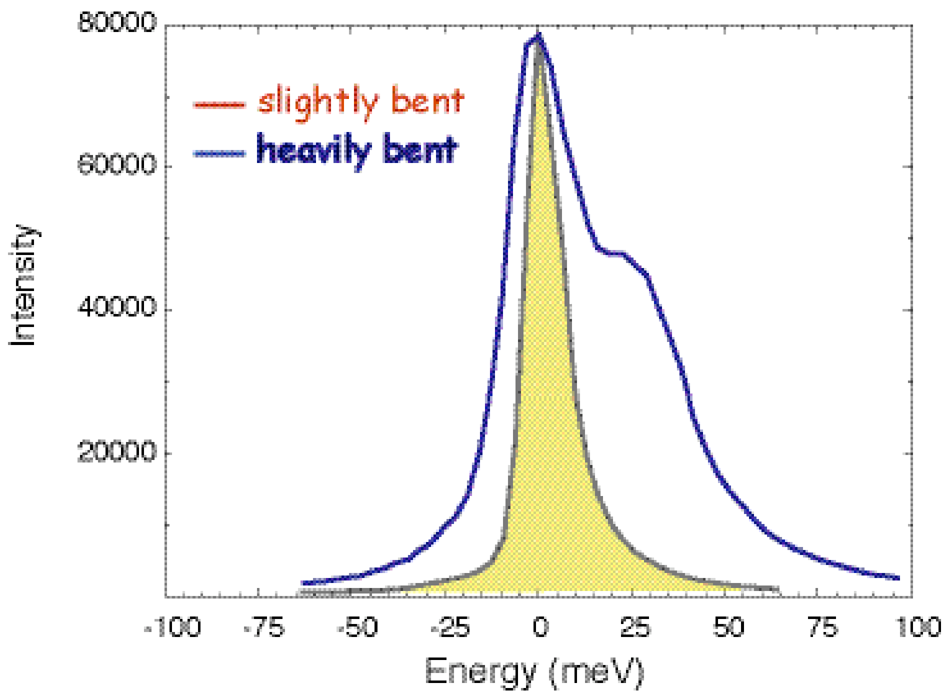
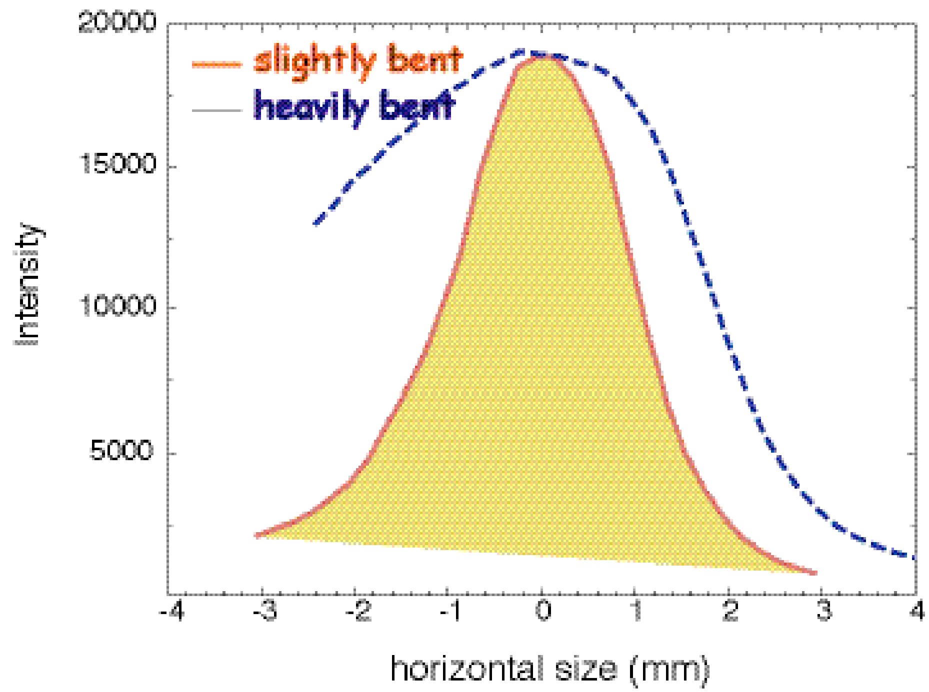
vertical

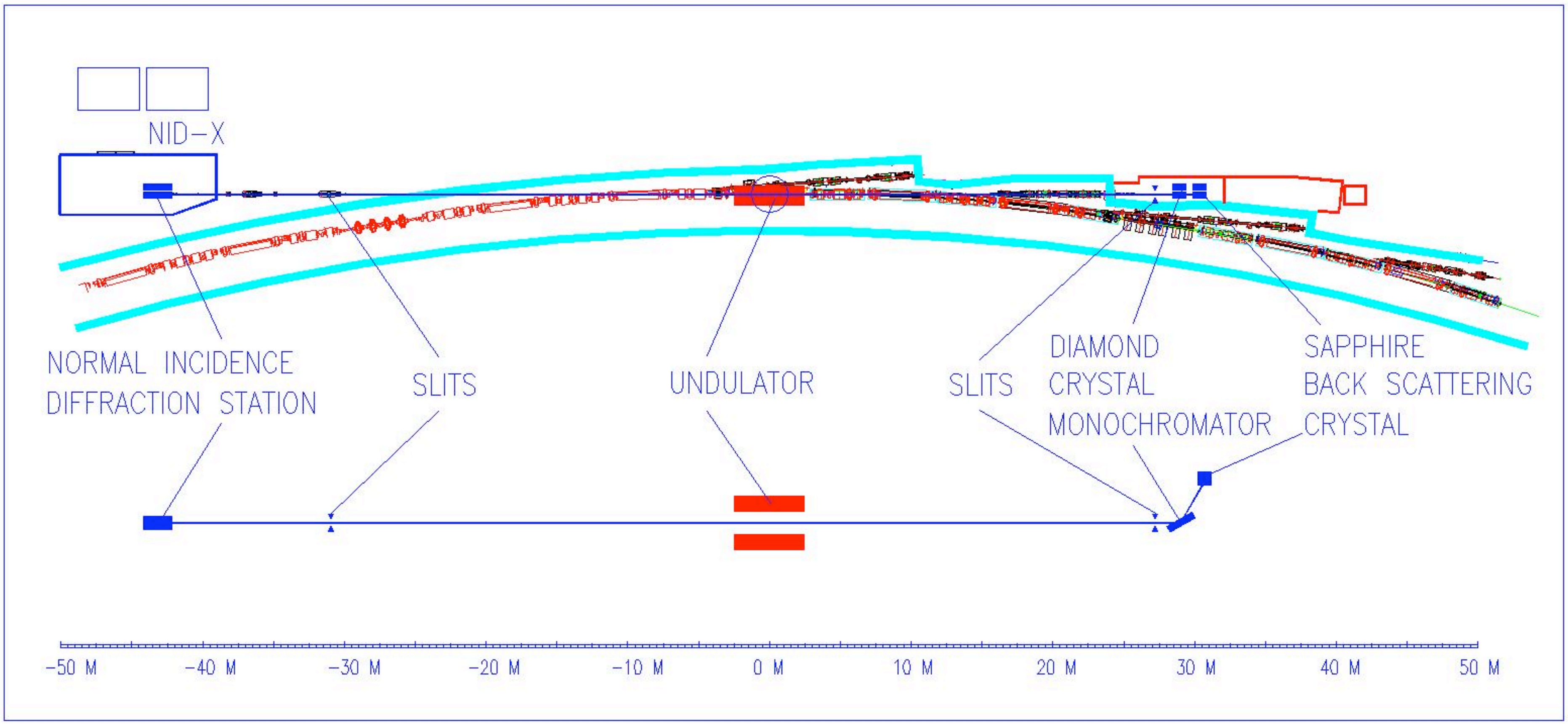


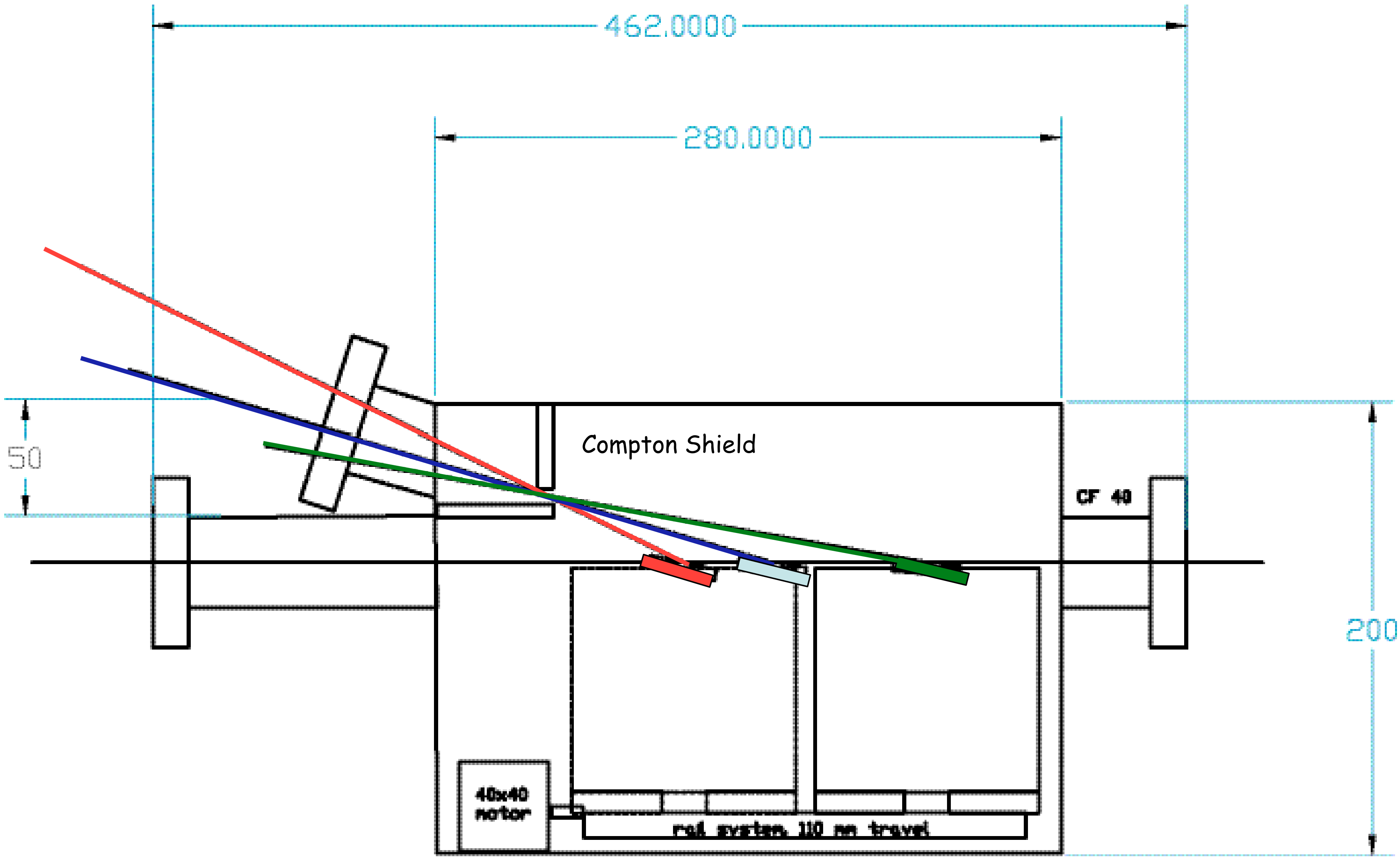
- beam size at mirror : 1.5mm
- focal spot size : <100µm
- mirror length : > E/keV * 0.04m

Effect of bending of an undiced, asymmetrically-cut back-reflecting crystal Si (12 4 0) on energy bandpass, and size of the beam at the NID-X station

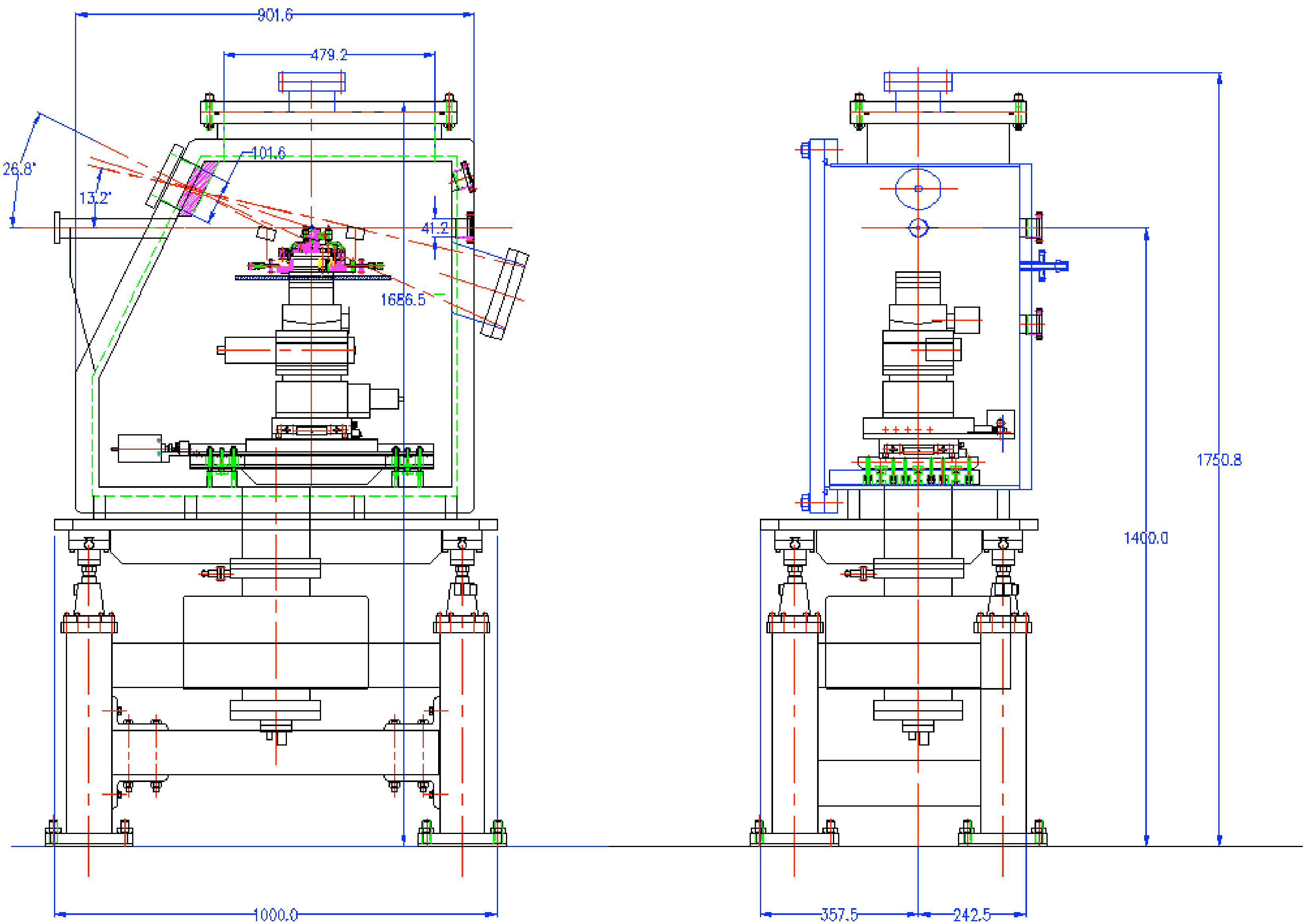




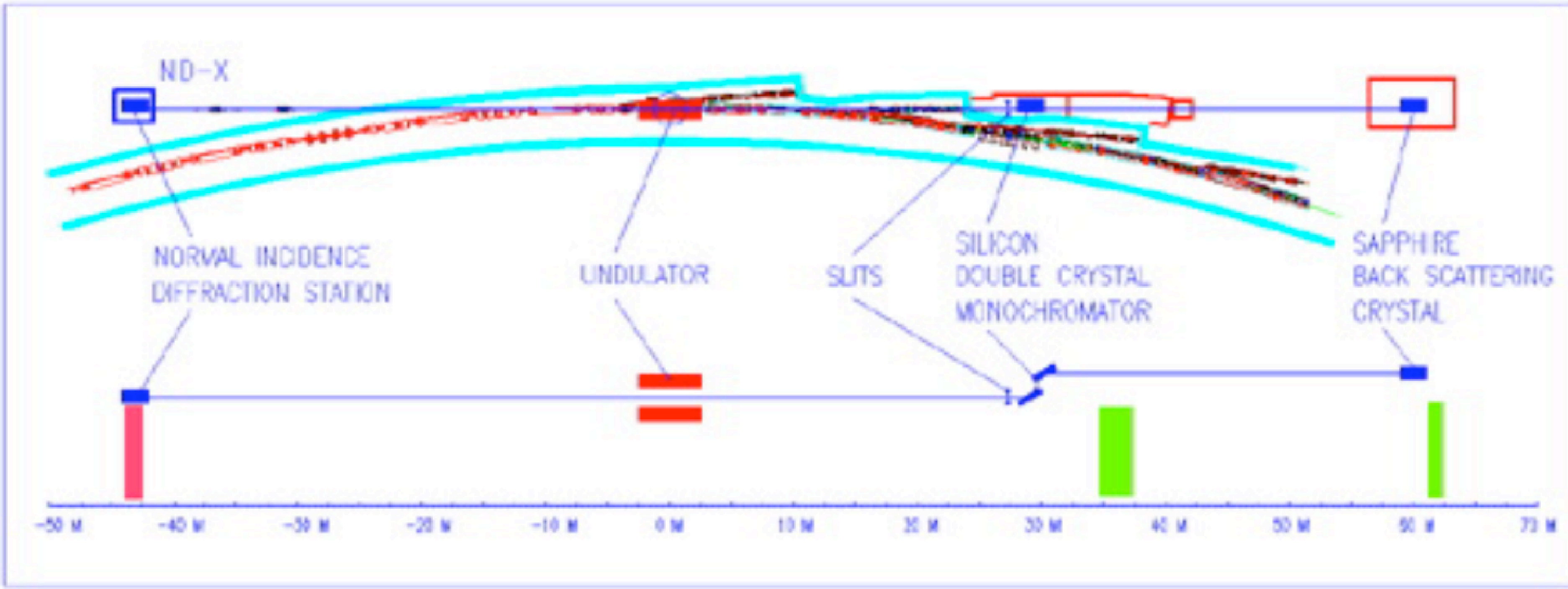

NID-X
NORMAL INCIDENCE
DIFFRACTION STATION
SLITS
UNDULATOR
SLITS
DIAMOND
CRYSTAL
MONOCHROMATOR
SAPPHIRE
BACK SCATTERING
CRYSTAL
-50 M
-40 M
-30 M
-20 M
-10 M
0 M
10 M
20 M
30 M
40 M
50 M



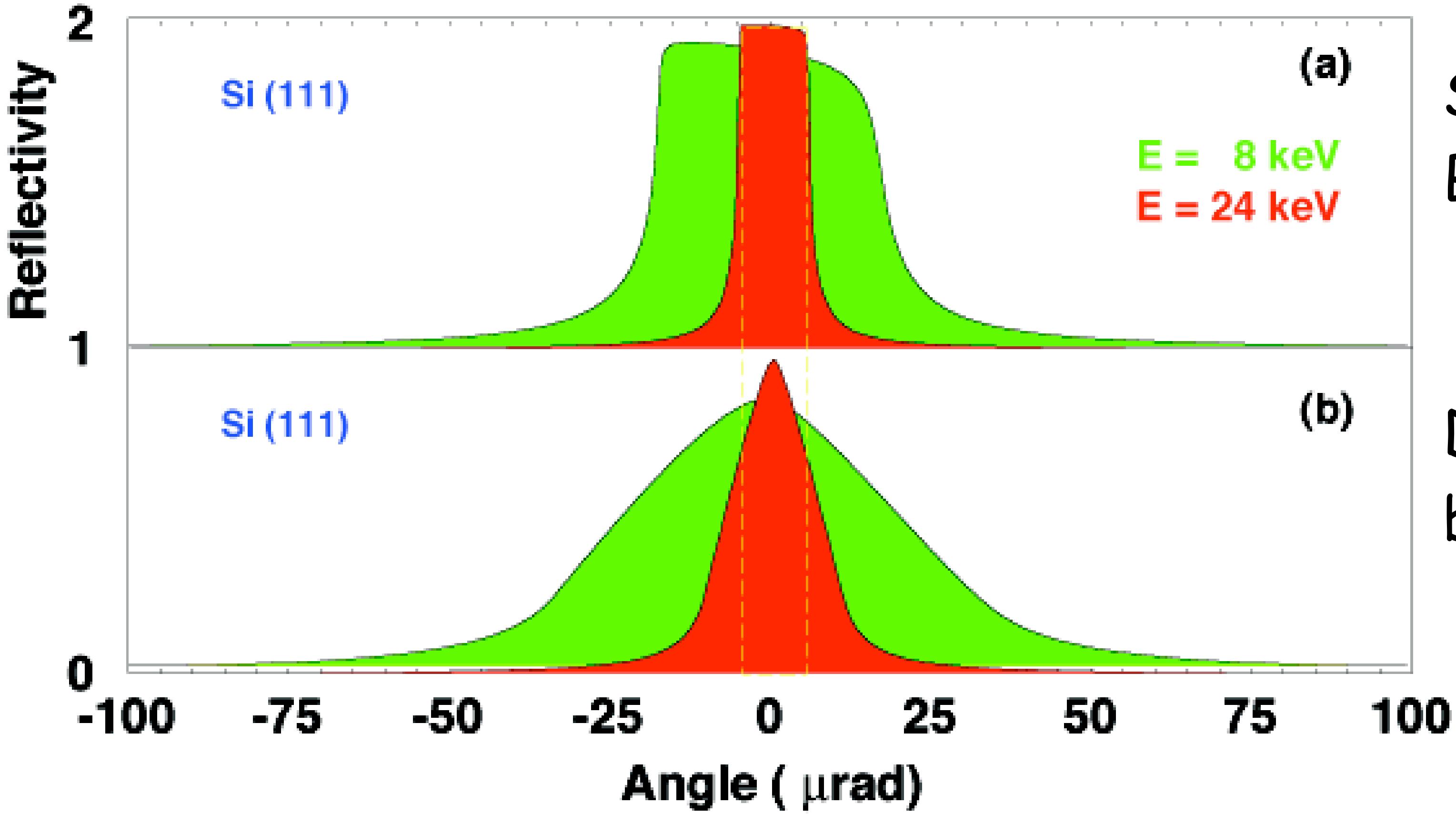
462.0000
280.0000
50
Compton Shield
CF 40
200
40x40
motor
rod system, 110 mm travel



X5-21 CRYSTAL MONOCHROMATOR FOR APS 1-ID-NID BEAMLINE


ND-X
NORMAL INCIDENCE
DIFFRACTION STATION
UNDULATOR
SLITS
SILICON
DOUBLE CRYSTAL
MONOCHROMATOR
SAPPHIRE
BACK SCATTERING
CRYSTAL

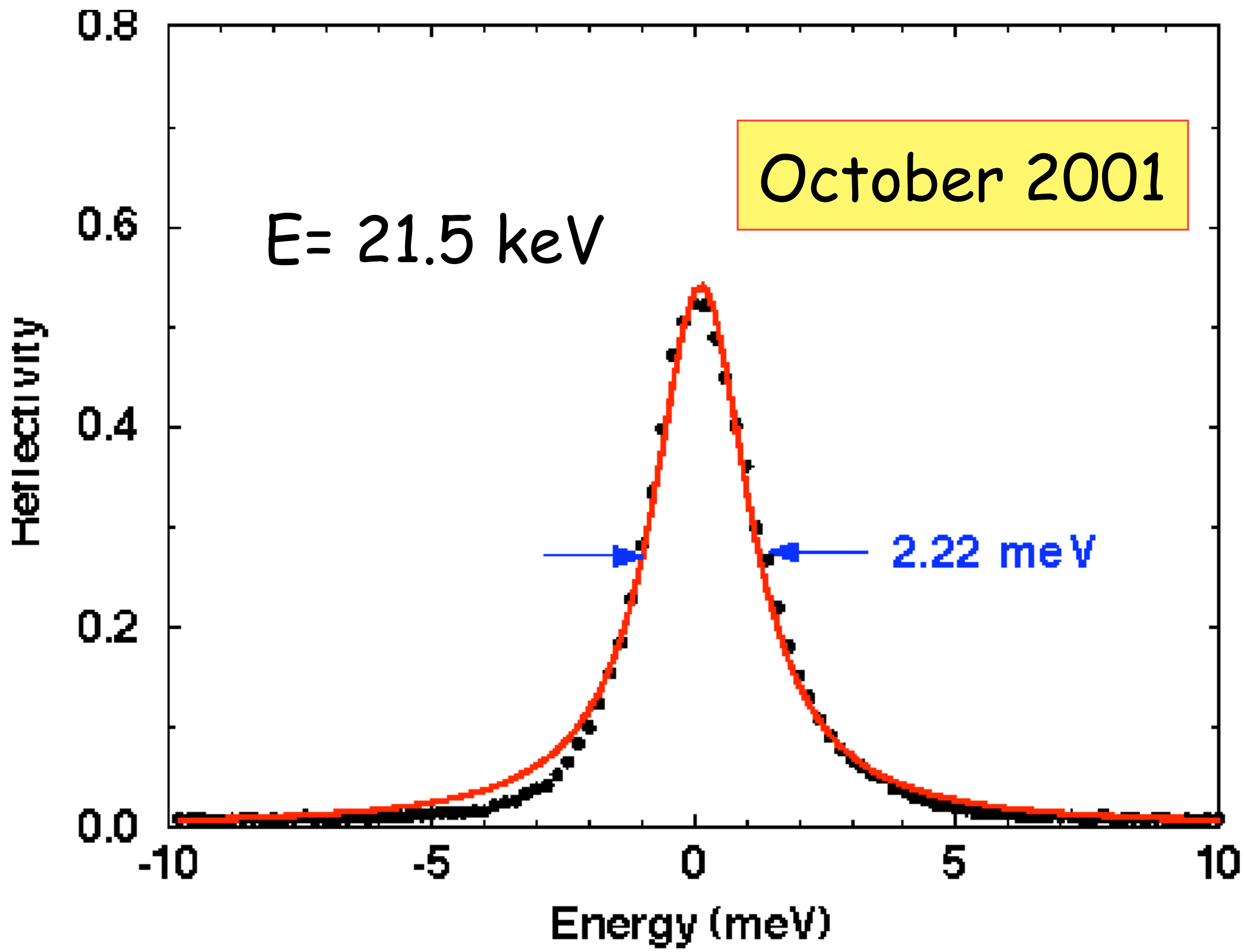
Limited vertical acceptance



Single Bounce

Double bounce

Does it work ?



Yes, it does work !

Observed reflections and flux at NID

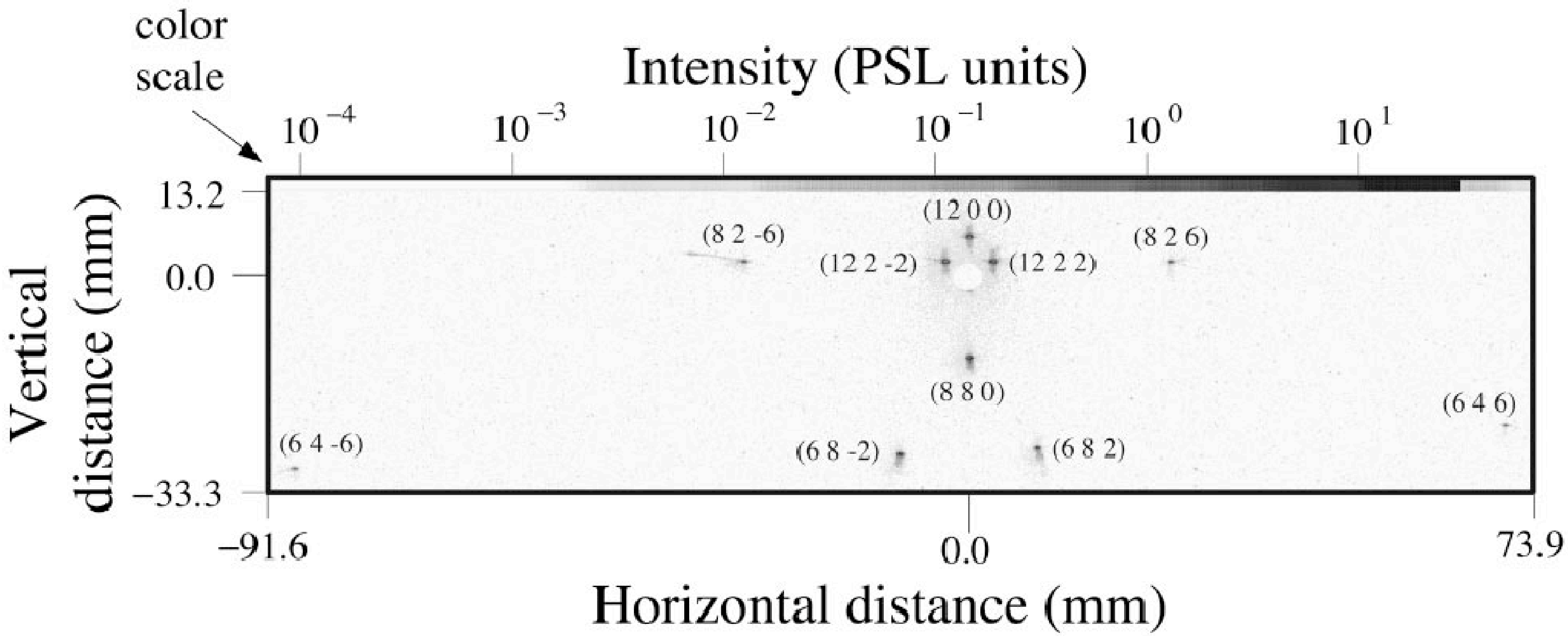
(h k l)	E (keV)	ΔE (meV)	Measured Flux (Hz)	Reflectivity
Si				
7 7 7	13.839	5.1	6×10^{9}	10-20 %
12 4 0	14.315	6.2		
8 8 8	15.816	4.4	5×10^{9}	
9 9 9	17.793	2.0	7×10^{8}	
Al_2O_3				
0 0 18	8.589	35.4	1.1×10^{11}	
1 0 22	10.604	2.5	1.7×10^{10}	
0 0 30	14.310	13.5	1.6×10^{10}	60-90%
1 2 29	14.399	1.7	8.5×10^{9}	
0 0 36	17.716	2.8	4.8×10^{9}	
0 0 42	20.045	1.8	2.5×10^{9}	
8 8 0	20.841			
9 9 0	23.446			
10 10 0	26.051			
11 11 0	28.656			
12 12 0	31.261			
13 13 0	33.866			
14 14 0	36.47			
15 13 14	37.109		$\sim 10^{7}$	

The spectral flux (i.e. flux/eV) obtained with sapphire is the highest available to date !

Multiple-beam x-ray diffraction near exact backscattering in silicon

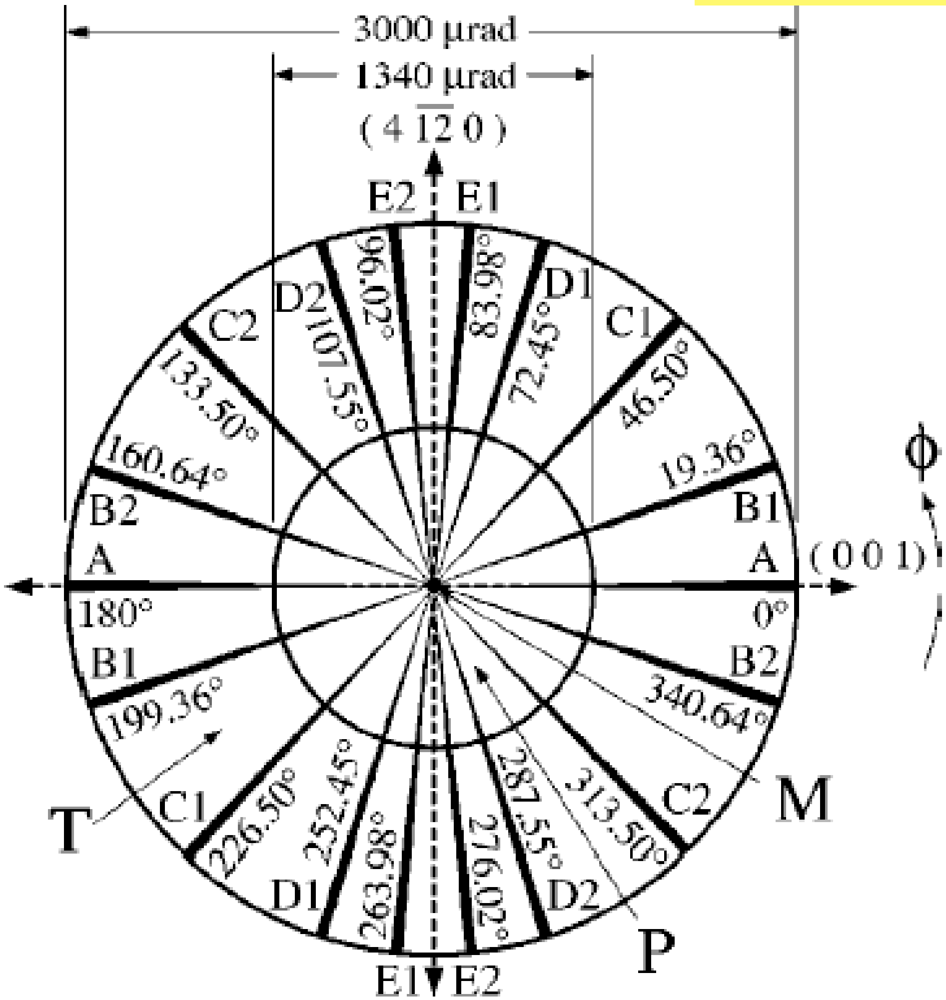
J. Sutter et al, Phys. Rev. B, 63 (2001) 94111

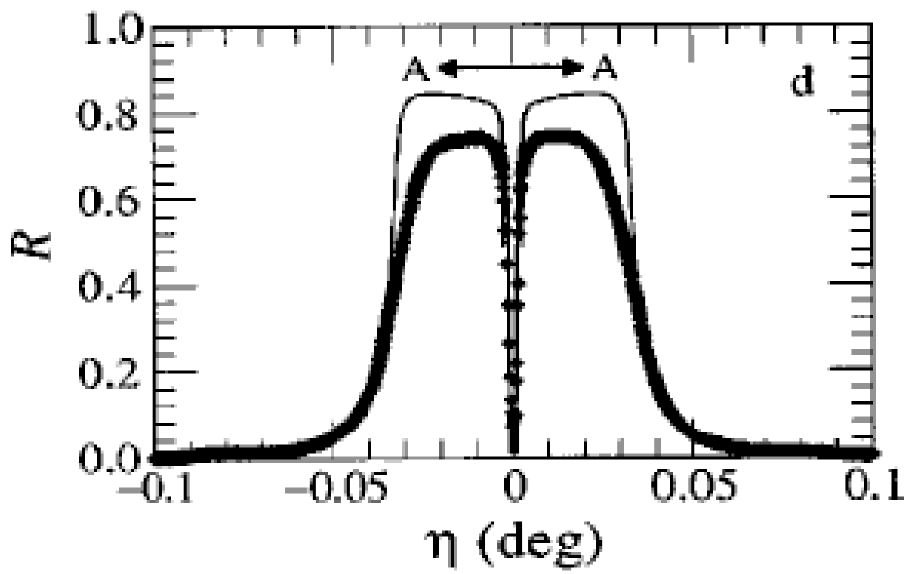
h,k,l= (12, 4, 0), E= 14. 315 keV



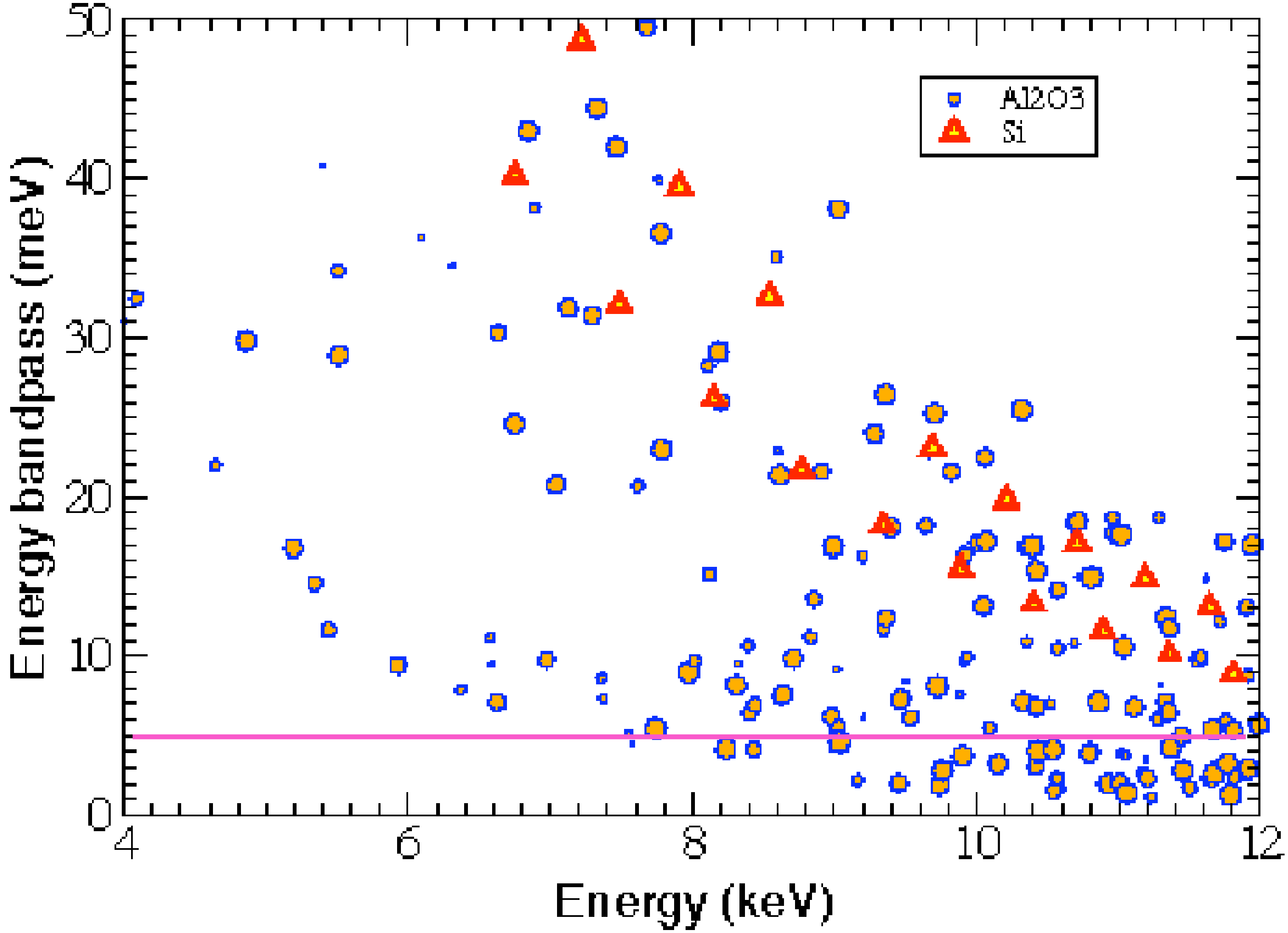
$$h^2 + k^2 + l^2 = hH + kK + lL$$

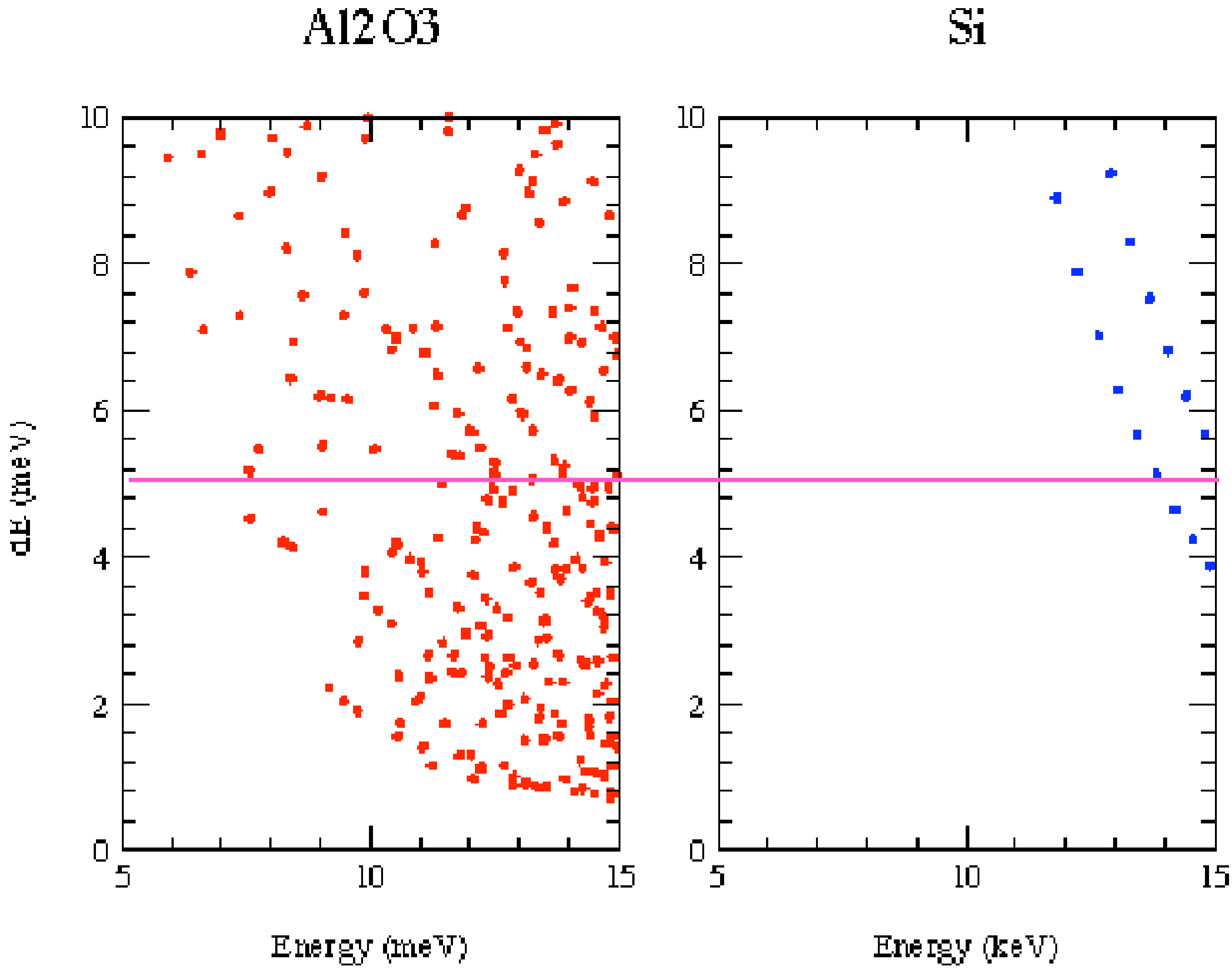
$$h^2 + k^2 + l^2 = hH + kK + lL$$





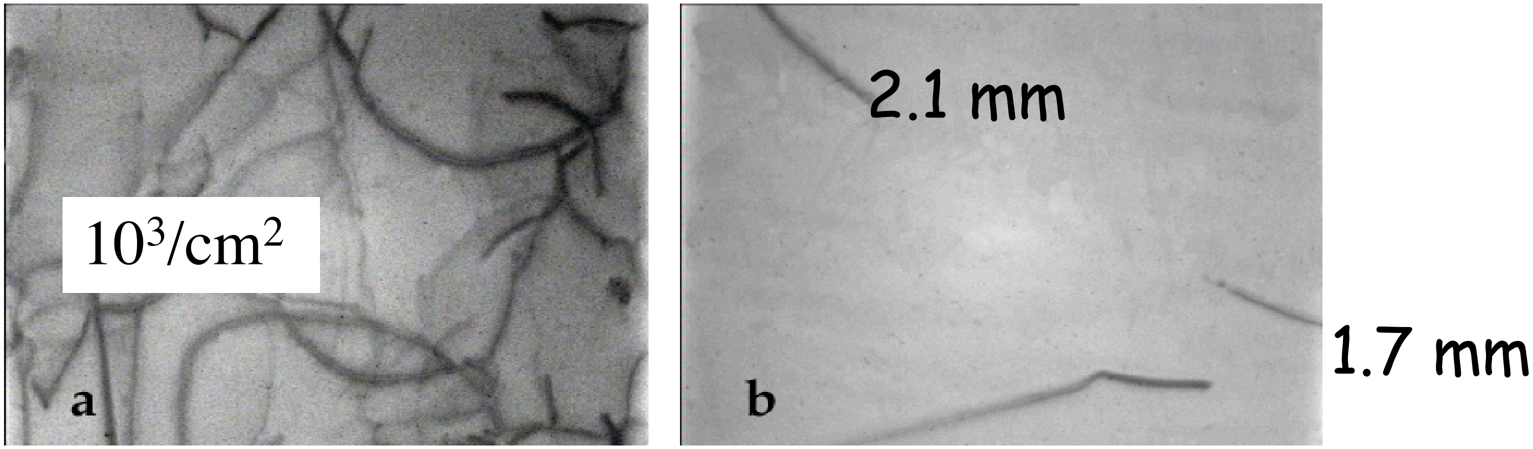
Why sapphire ?

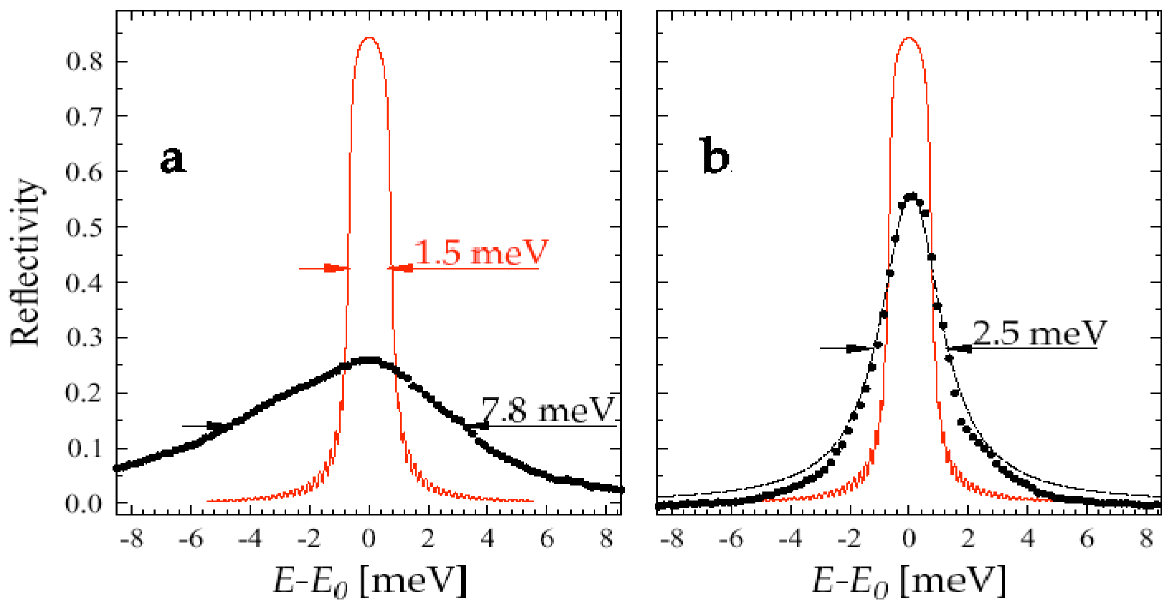




Al2O3
Si
dE (meV)
Energy (meV)
Energy (keV)
0
2
4
6
8
10
5
10
15

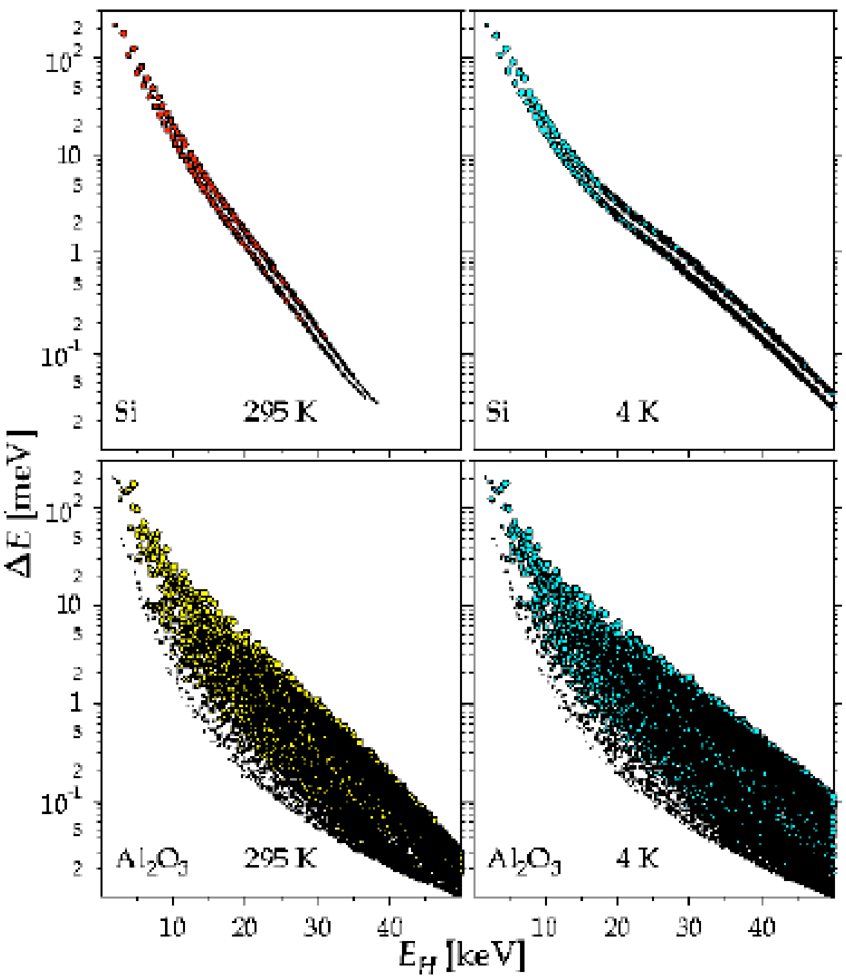
Why not sapphire ?

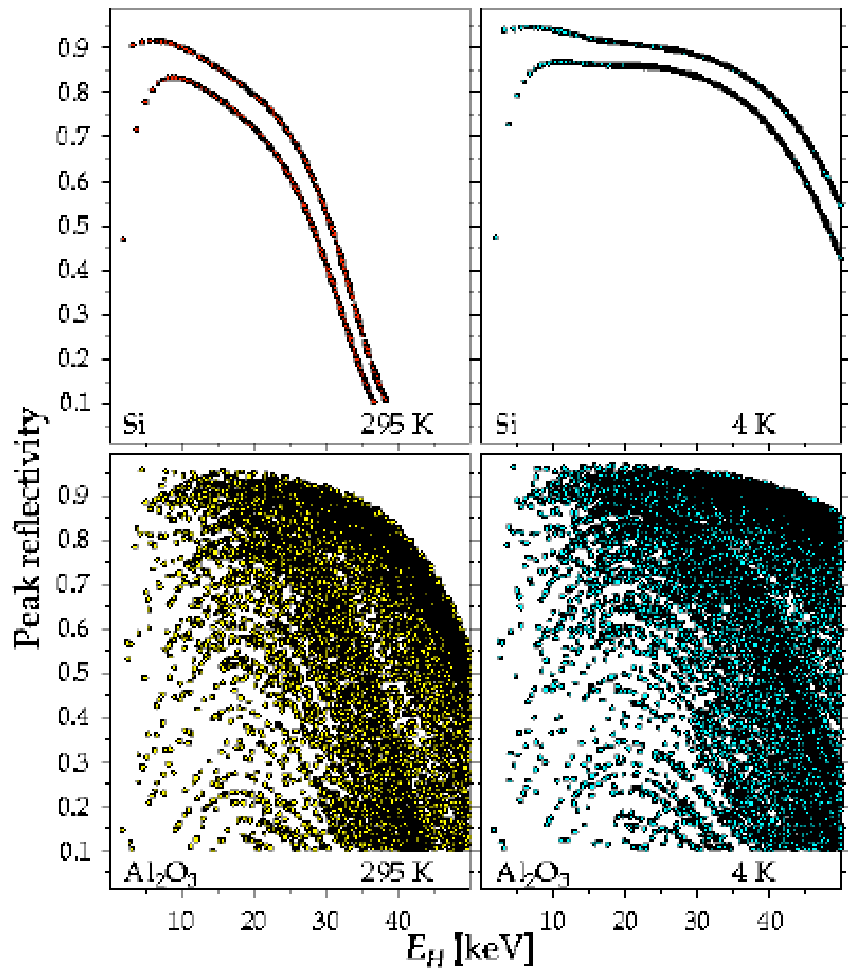




E=21.5 keV

Why sapphire ?





Shvydko, et al (2002)

Intrinsic scientific advantages

- Access to high energy resolution
  - at lower energies (5 < E < 15 keV)
  - at many more energies (every ~15-to-150 eV)
  - at specific energies critical for RIXS
  - at higher energies (E > 30 keV)
  - with highest efficiency (R > 90 %)
- Longest beamline on the APS floor (up to 210 m)
  - Largest coherence volume
  - Smallest possible divergence
- Geometrically unique optics critical for IXS
  Suitable for an x-ray cavity

What is high resolution in the hard x-ray regime ?

Resolution power, $R = E/\Delta E$

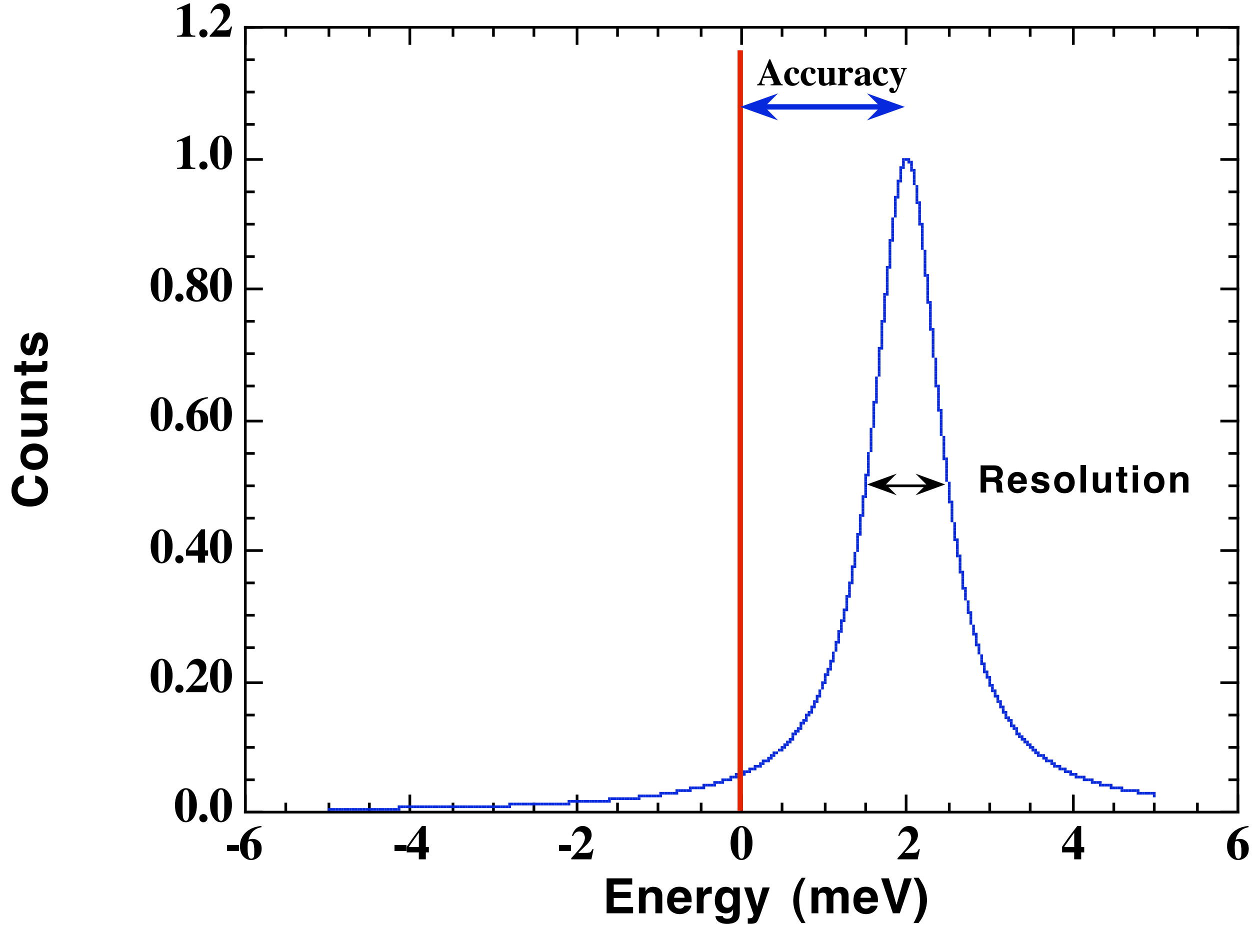
The proposed beamline is focused for the following energy and resolution range:

$$7 < E < 60 \text{ keV}$$

$$10^{-9} < \Delta E < 10^{-3} \text{ eV}$$

$$10^{7} < R < 10^{13}$$

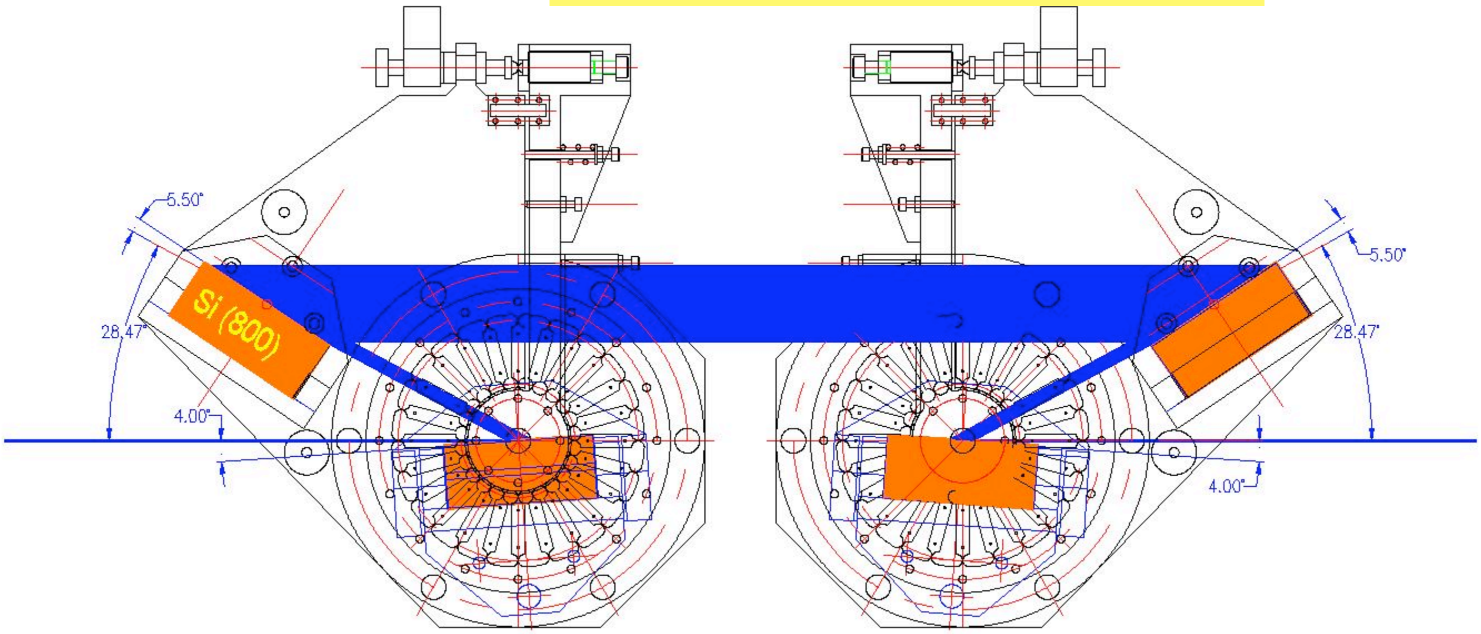
The proposed beamline NID at the APS is designed this energy range to study collective excitations in condensed matter and hyperfine interactions for nuclear resonance scattering, x-ray metrology, and coherent imaging.



1.2
1.0
0.80
0.60
0.40
0.20
0.0
Counts
Accuracy
Resolution
-6
-4
-2
0
2
4
6
Energy (meV)

New monochromators with artificially linked, dispersive channel-cut configuration

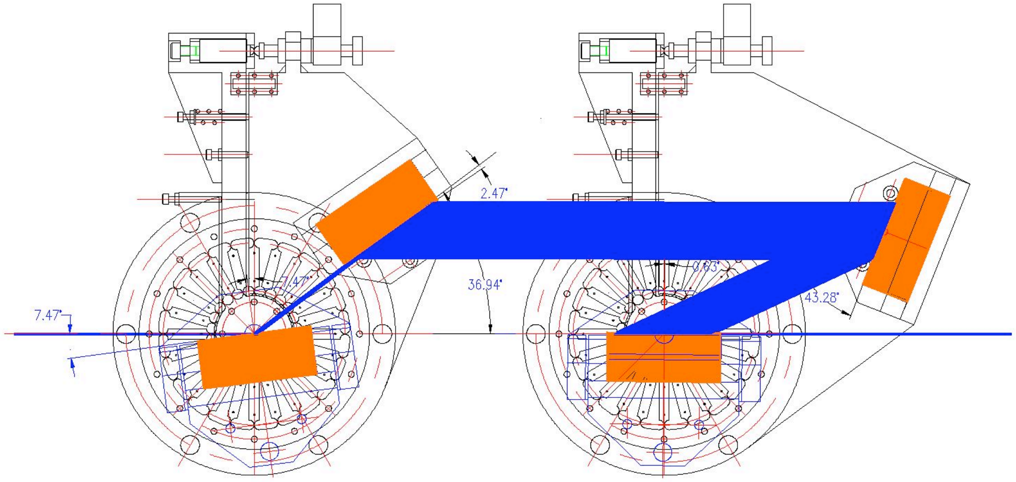
ΔE= 1 meV, E = 9.4 keV

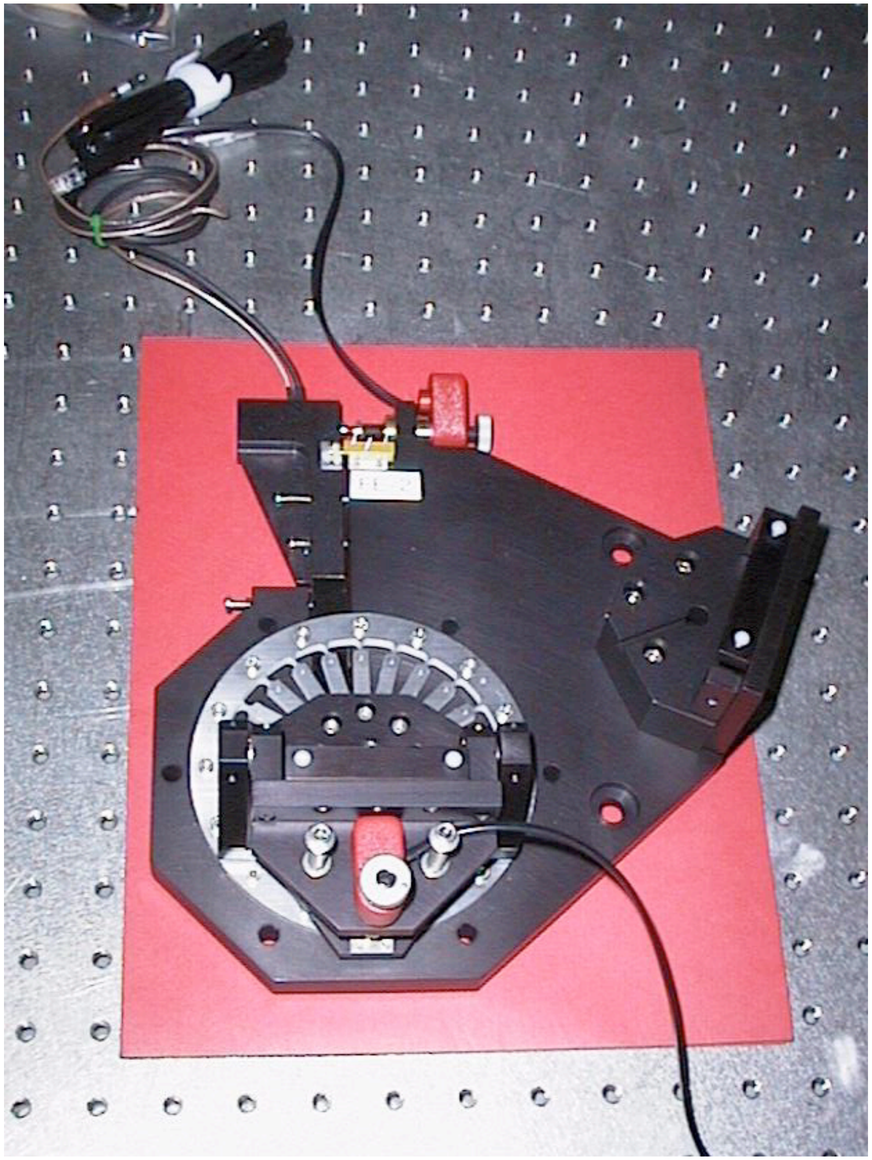


Deming Shu, Thomas Toellner, APS-ANL

sub-meV mono's between 9-30 keV

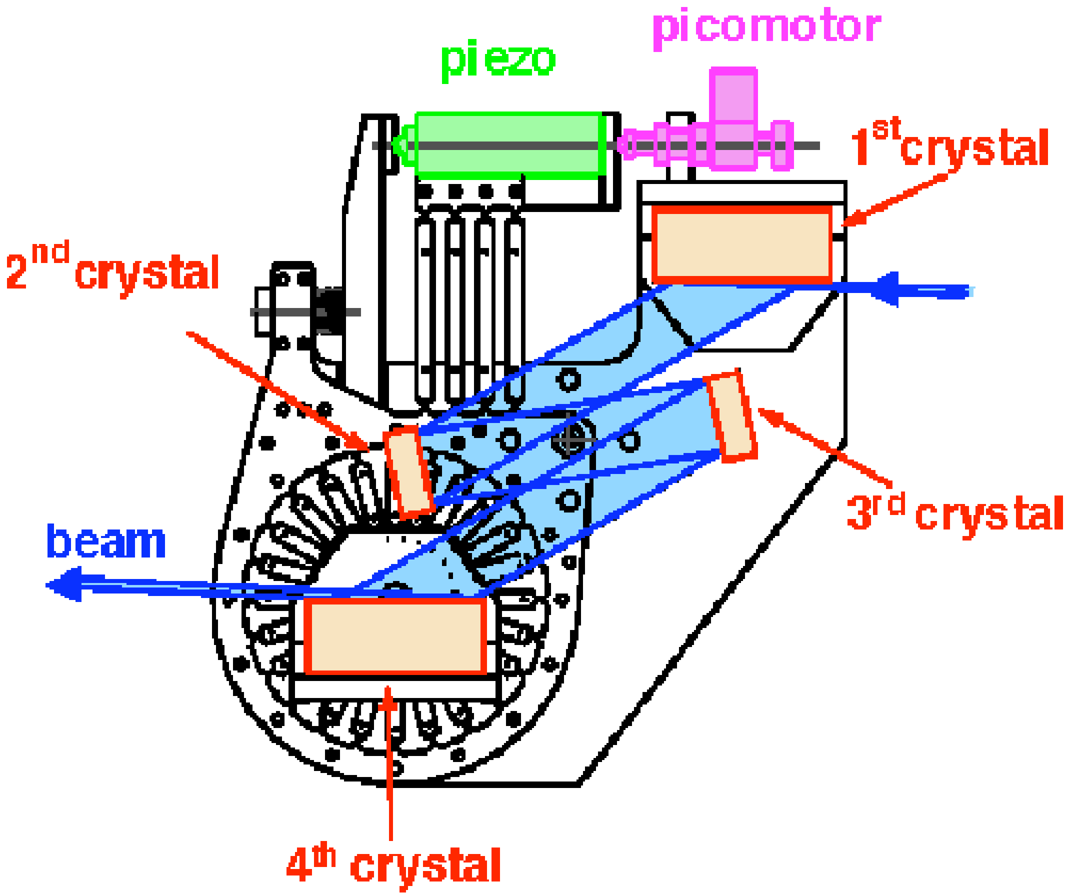
ΔE= 1 meV, E = 14.4 keV

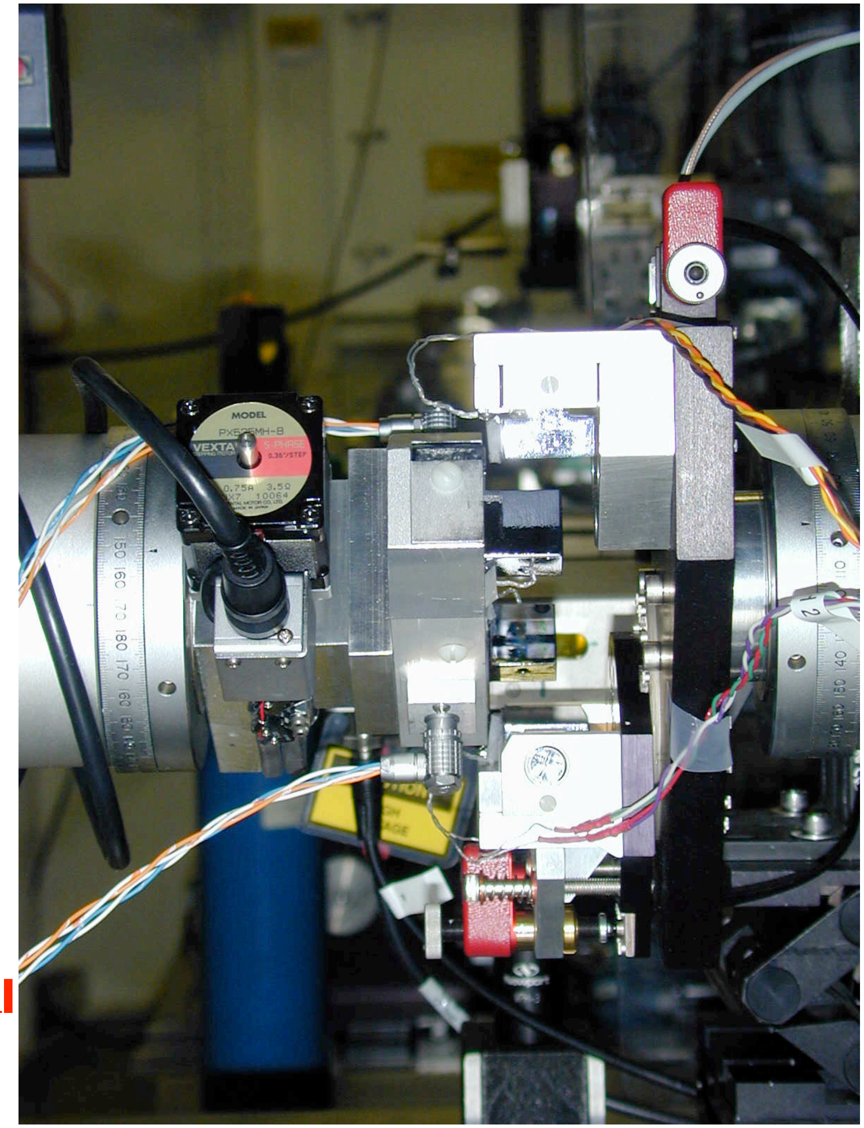




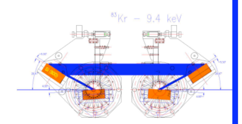
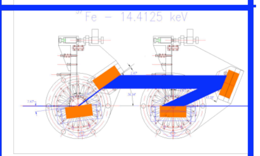
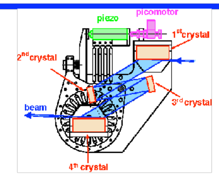
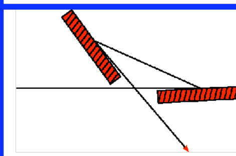
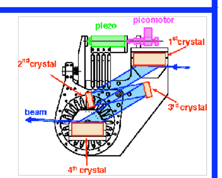
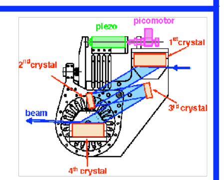
Artificially linked nested mono's

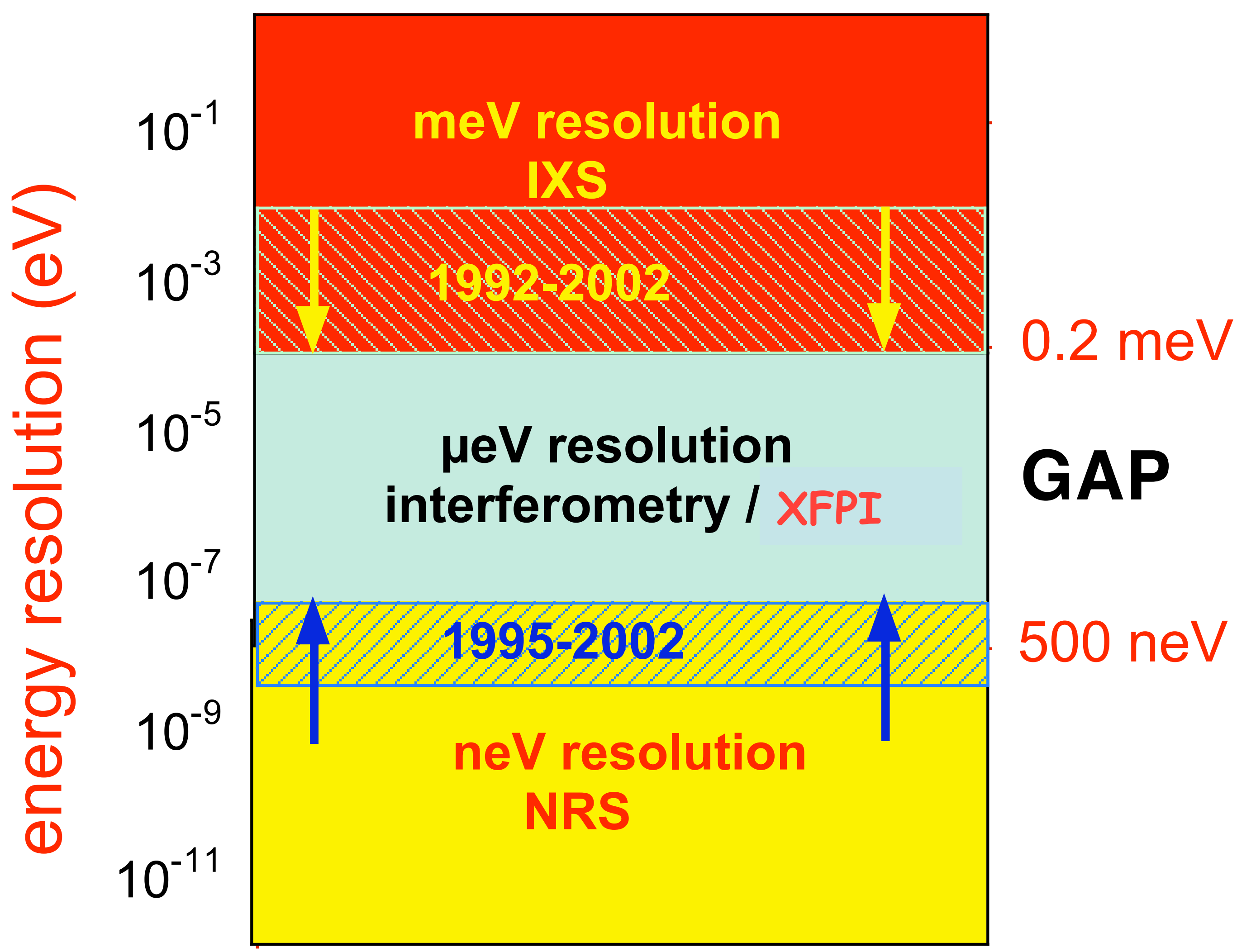
ΔE = 0.8 meV, E = 21.5 keV





Advances in high energy resolution monochromators in the new century at the APS , 3-ID beamline

Isotope	Energy (keV)	ΔE (meV)	Flux (GHz)	Type
^{83}Kr	9.4035	1.0	6	
^{57}Fe	14.4126	1.0	1.2	
IXS ^{151}Eu	21.65 21.54	0.7 0.8	0.8 0.4	
^{119}Sn	23.879	0.14	0.004	
^{119}Sn	23.879	0.85	0.2	
^{161}Dy	25.6514	0.5	0.1	



energy resolution (eV)
10-1
10-3
10-5
10-7
10-9
10-11
meV resolution
IXS
1992-2002
0.2 meV
μeV resolution
interferometry / XFPI
GAP
1995-2002
500 neV
neV resolution
NRS

Measuring wavelengths and lattice constants with Mossbauer wavelength standard

Higher accuracy ($\Delta E/E \sim 10^{-13}$ possible)

Reproducible independent of temperature, pressure, composition, and other parameters

Available between 6-100 keV range at more than a dozen energies

Y. Shvydko, et al, Phys. Rev.Lett., 45 (2000) 495
J. Synchrotron Rad. 9 (2002) 17

λ-meter

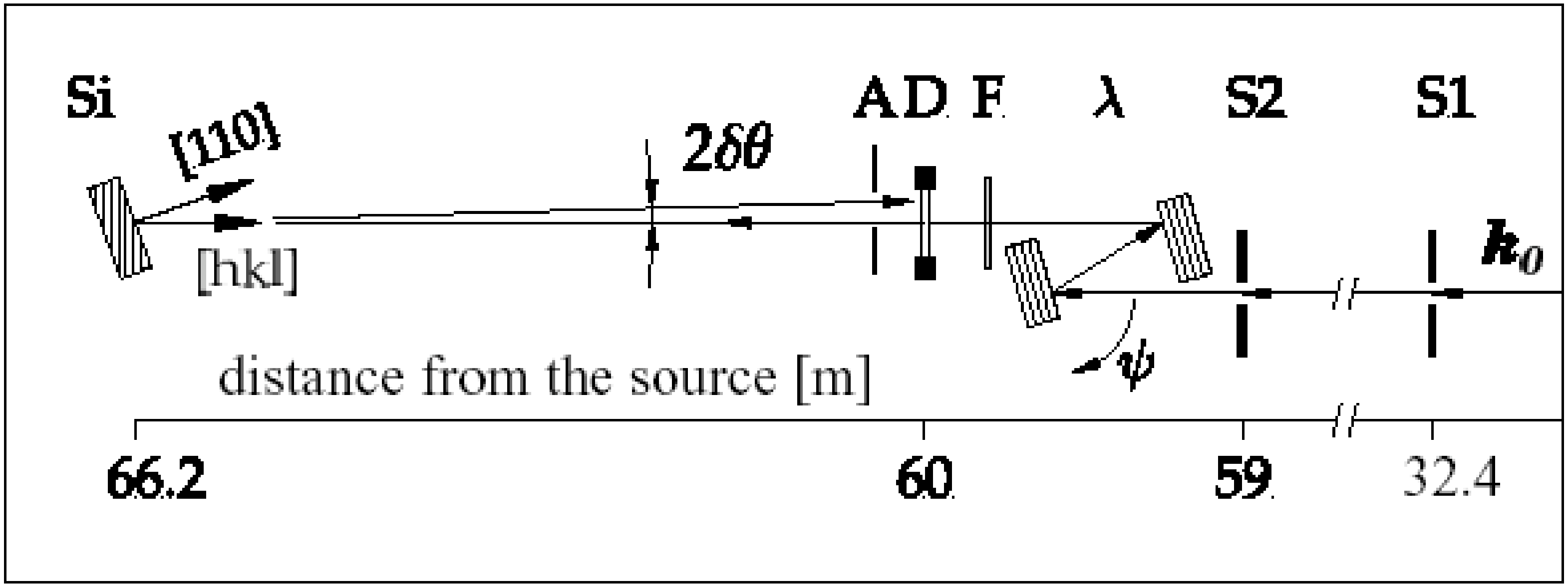
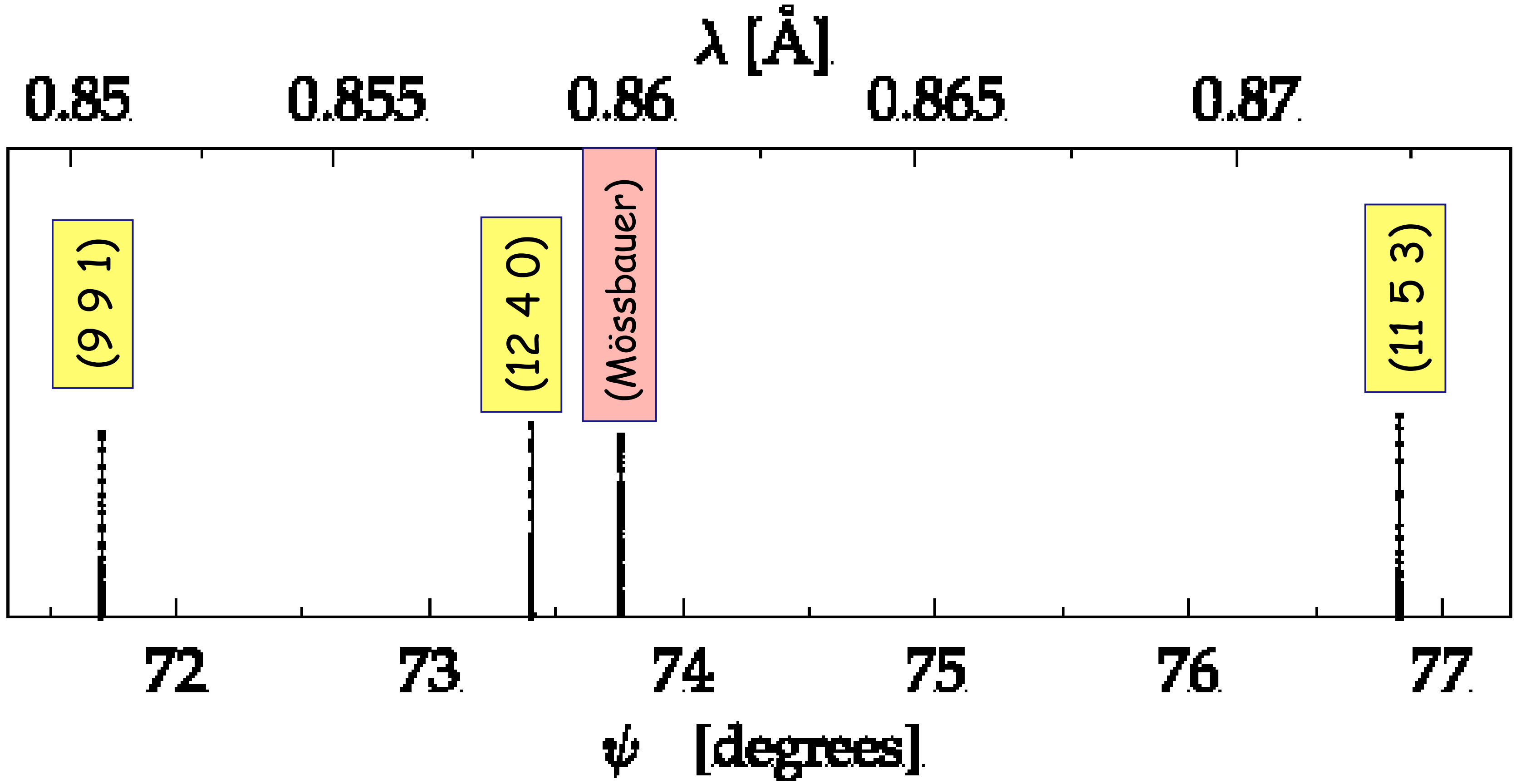
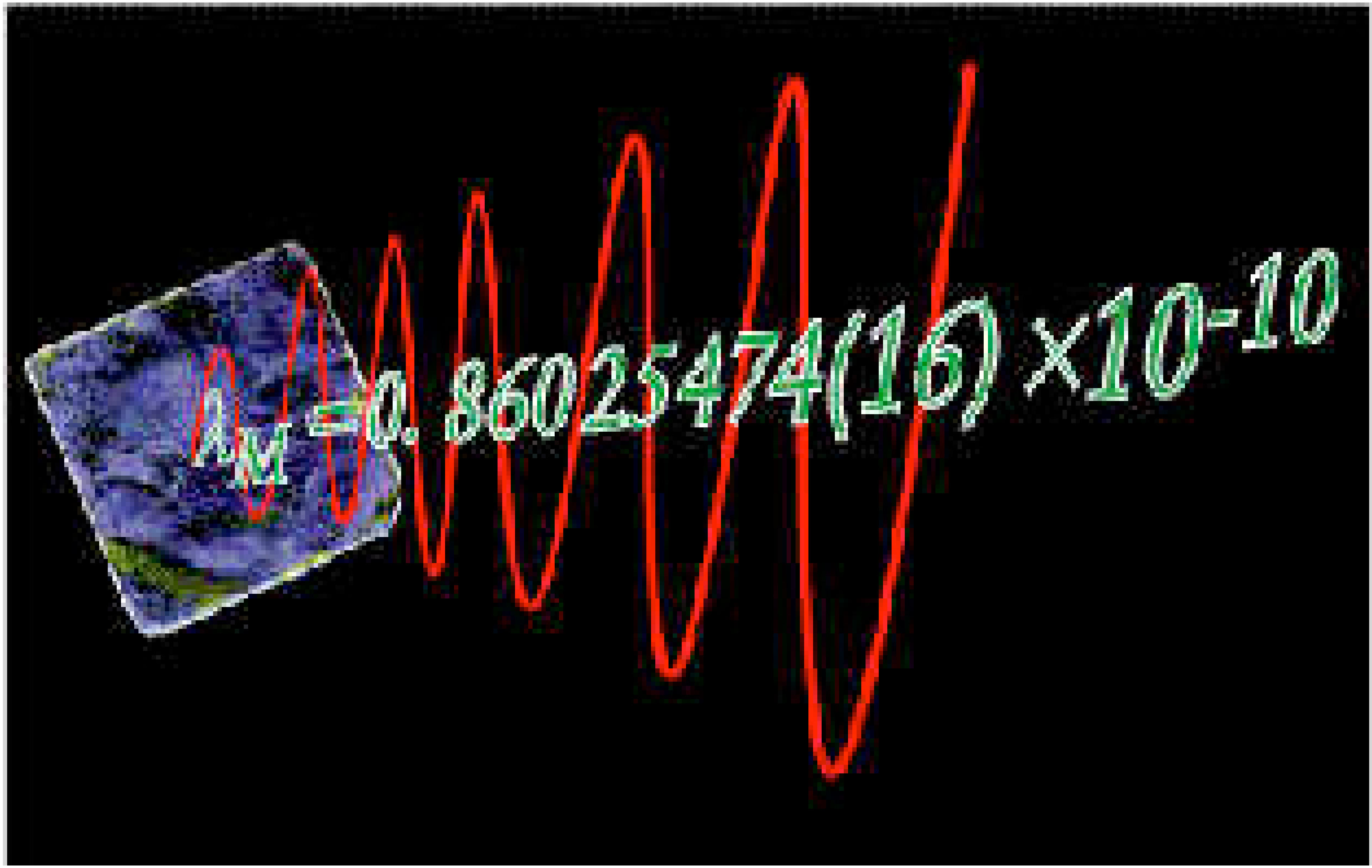


Figure 4.1: Schematic of the experiment: the radiation after a high-heat-load premonochromator (not shown) passes through the vertical slits S1 and S2, λ: λ-meter; F: ^{57}Fe foil used as a source of Mössbauer radiation; D: semitransparent avalanche photo-diode with 1 ns time resolution; A: 4 mm aperture, Si: reference silicon single crystal with (110) surface in an evacuated thermostat on a 4-circle goniometer. The distances from the APS source are given.

Phys. Rev.Lett., 45 (2000) 495

Calibration against Si lattice constant




λM =0. 86025474(16) ×10-10

Wavelength & energies of Mossbauer isotopes determined at a synchrotron radiation source

isotope	E (eV)	□ (Å)	□□/□ (10^{-7})
^{57}Fe	14412.497(3)	0.86025474(16)	1.9
^{151}Eu	21541.418(10)	0.57556185(27)	4.7
^{119}Sn	23879.478(18)	0.51920811(39)	7.4
^{161}Dy	25651.368(10)	0.48334336(19)	4.0

How to measure the lattice constant and thermal expansion coefficient ?

Measure the wavelength of diffracted beam as a function of temperature accurately.

$$\lambda=2d$$

$$\Delta E/E_0=-\Delta d/d$$

Bragg back-scattering

Isotopic dependence of lattice constants

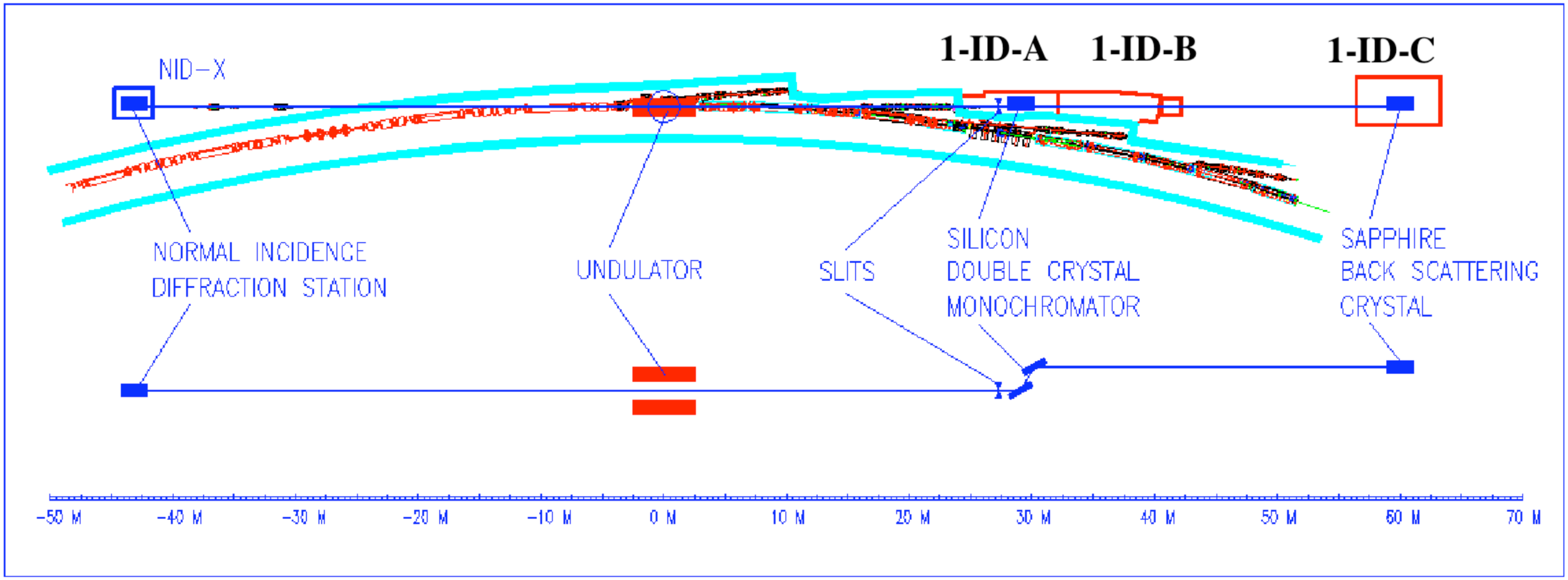
- Two crystals made up of same chemical element and different isotopes would have different inter-atomic distance (1958, H. London, Z, Phys. Chem. 16 (1958) 302).

- This effect is most significant in "quantum solids" like He.

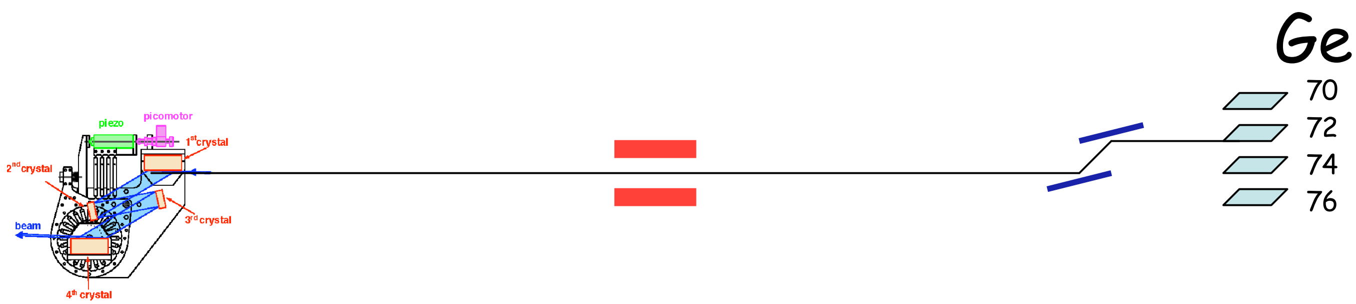
- Measurement of lattice constants and their temperature dependence provides information on "anharmonic" nature of bonding, "ZERO-POINT MOTION", as well as any subtle phase transformation.

- The difference in inter-atomic distance between different isotopes can be as large as a few parts in 10,000. But, typically this number is parts per million or smaller for heavier elements.

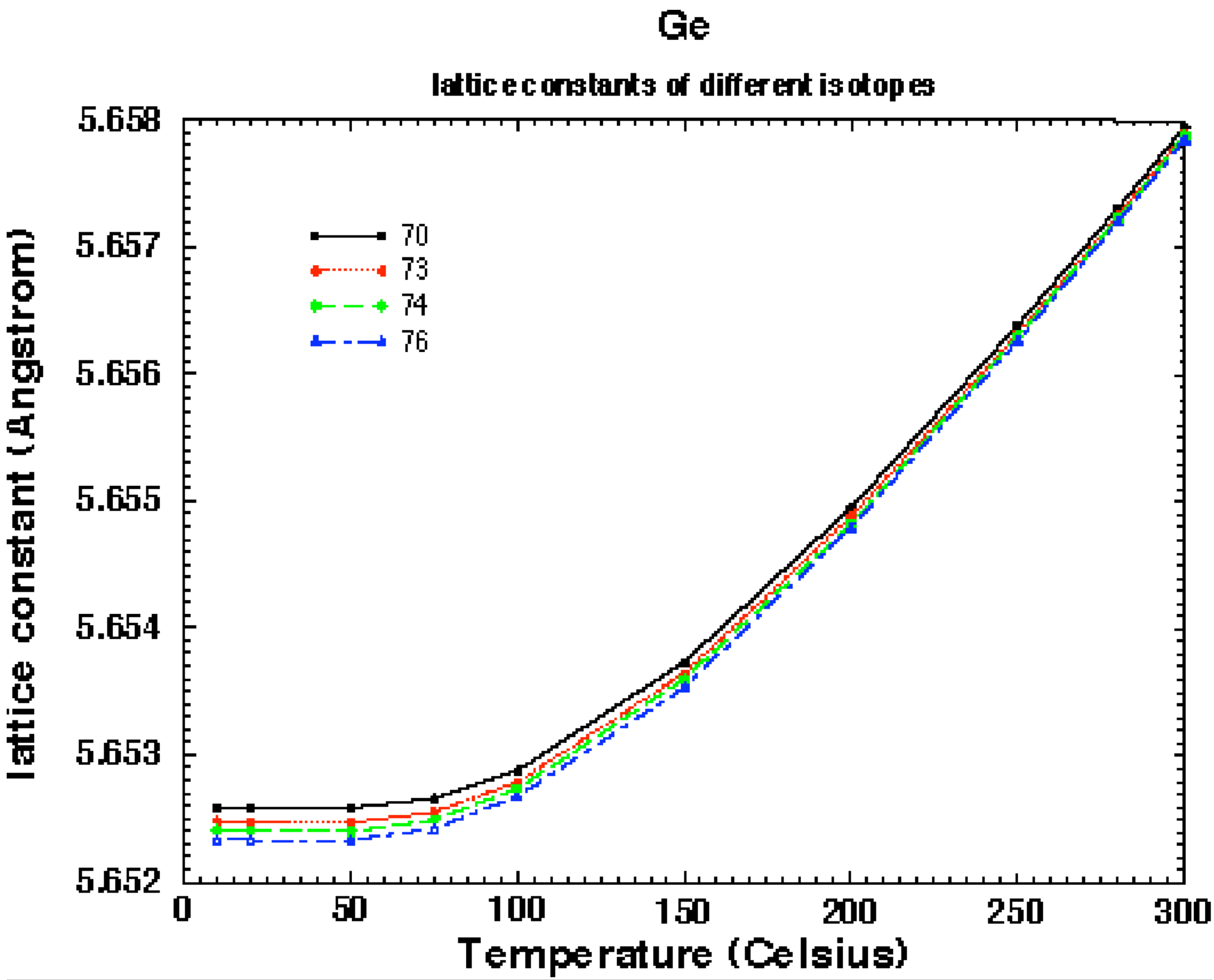
- Real thermal expansion coefficient or Gruneisen constant can be measured and compared to theory only for isotopically pure materials.



NID–X
1-ID-A
1-ID-B
1-ID-C
NORMAL INCIDENCE DIFFRACTION STATION
UNDULATOR
SLITS
SILICON DOUBLE CRYSTAL MONOCHROMATOR
SAPPHIRE BACK SCATTERING CRYSTAL
–50 M
–40 M
–30 M
–20 M
–10 M
0 M
10 M
20 M
30 M
40 M
50 M
60 M
70 M



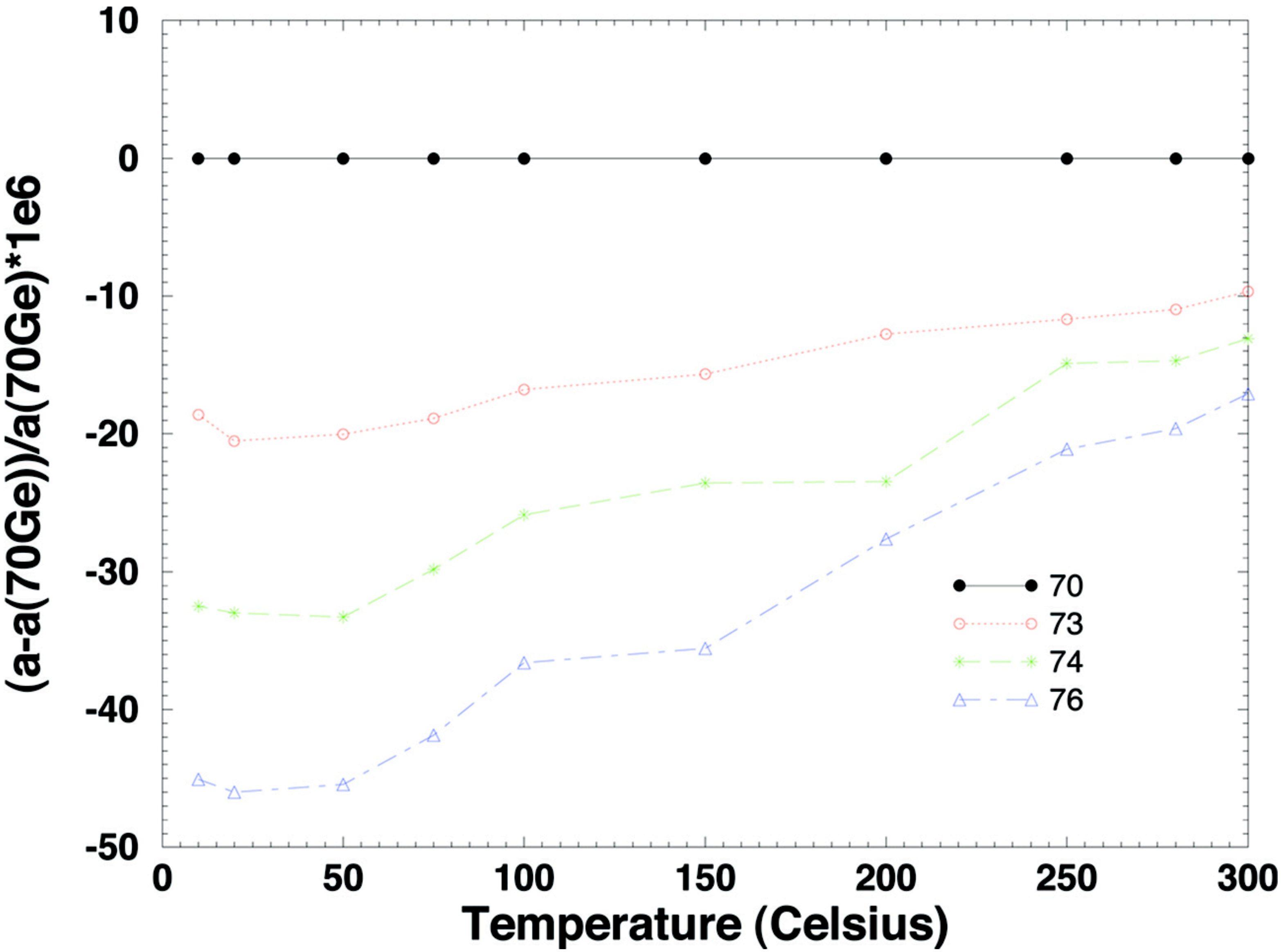
Ge
70
72
74
76
piezo
picomotor
1st crystal
2nd crystal
3rd crystal
4th crystal
beam

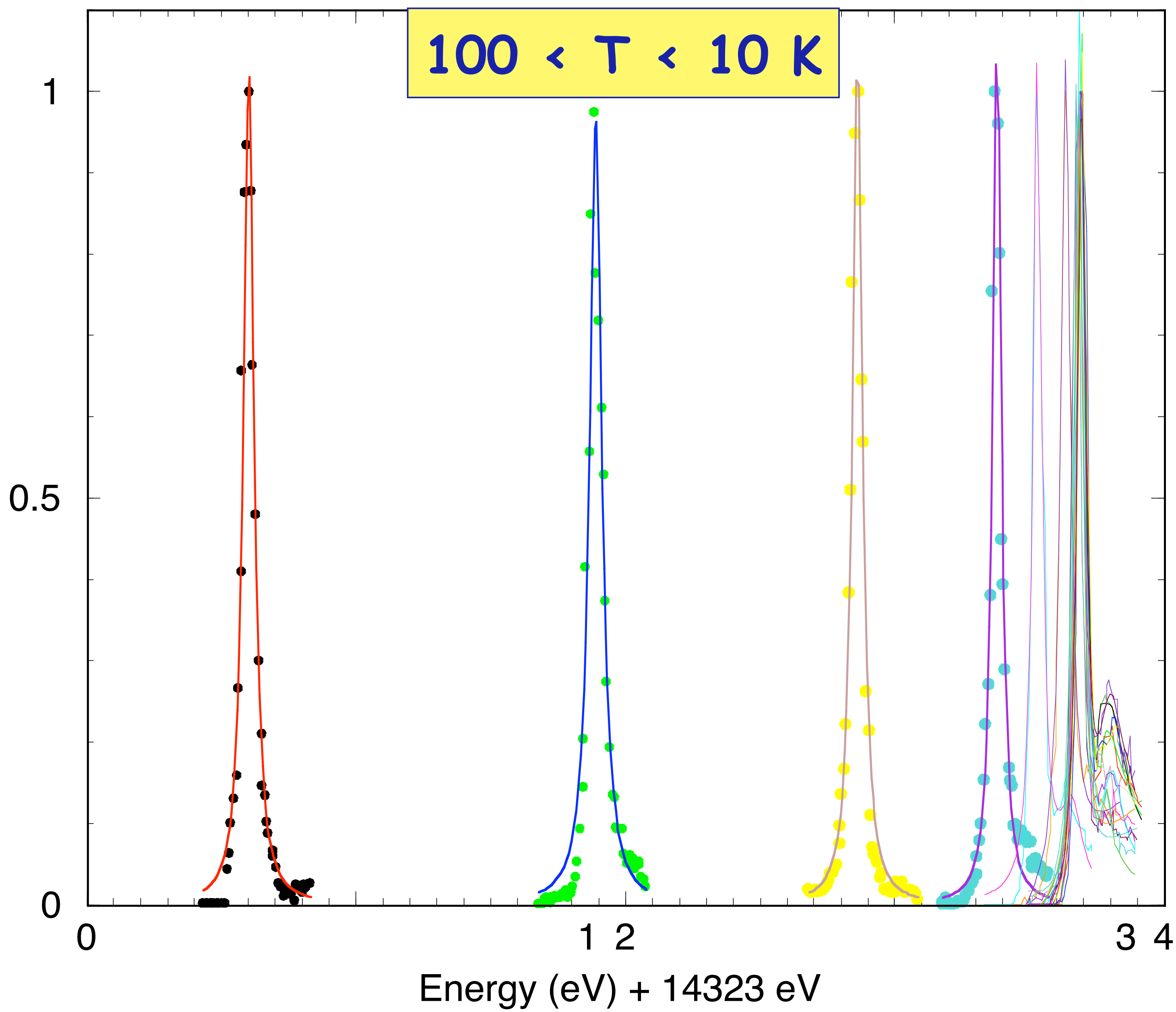

Ge
lattice constants of different isotopes
lattice constant (Angstrom)
Temperature (Celsius)
70
73
74
76
5.658
5.657
5.656
5.655
5.654
5.653
5.652
0
50
100
150
200
250
300

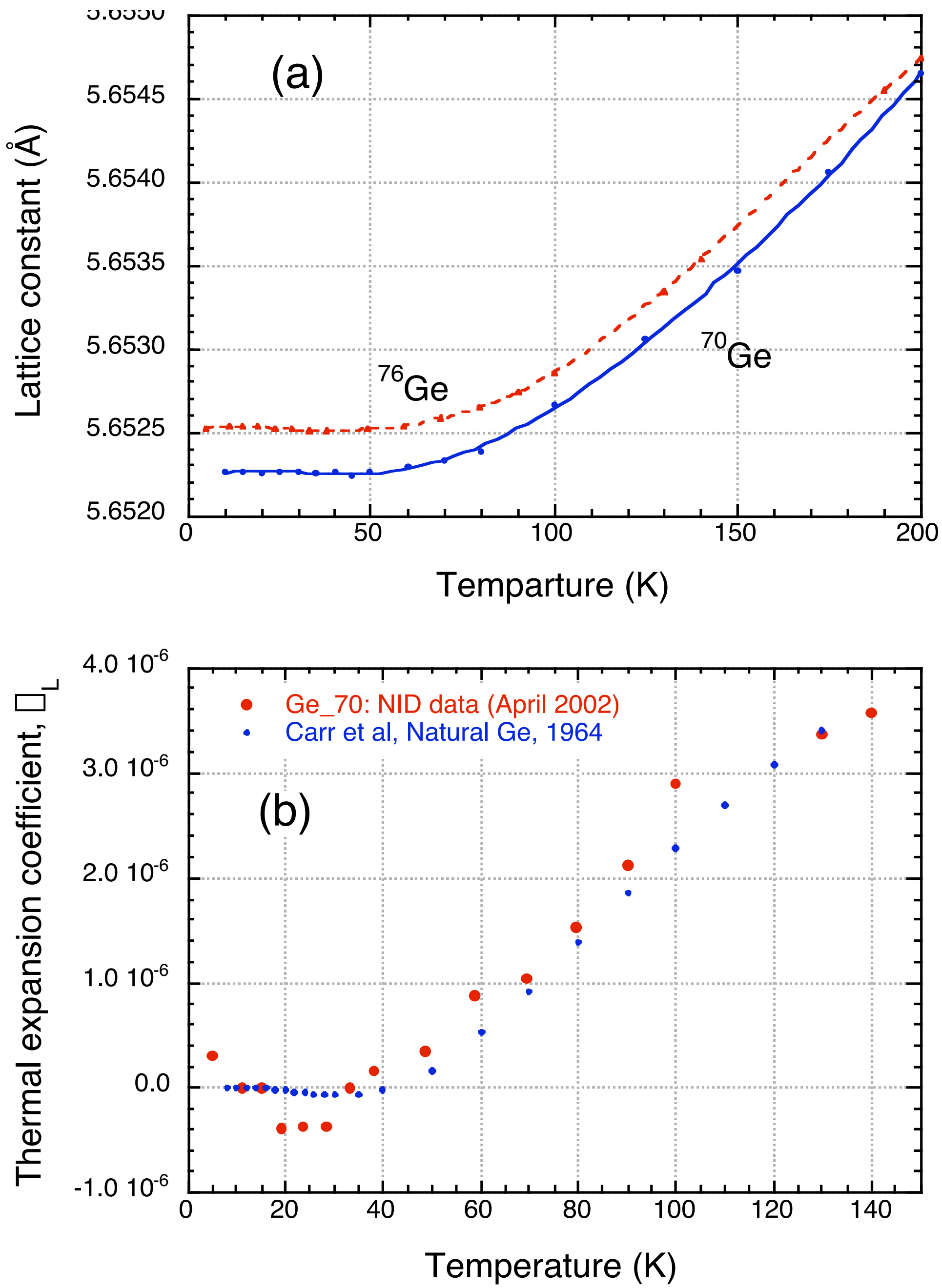
Lattice constants of Ge-isotopes

M	a_0(Å)	T(K)	M (amu)
70	5.652521	8-21	70.04953
73	5.652421	8-60	72.90906
74	5.652336	10.3-10.6	73.85475
76	5.652267	9.8	75.38534

M. Hu, et al, Phys. Rev. B (67) 113306 (2003)

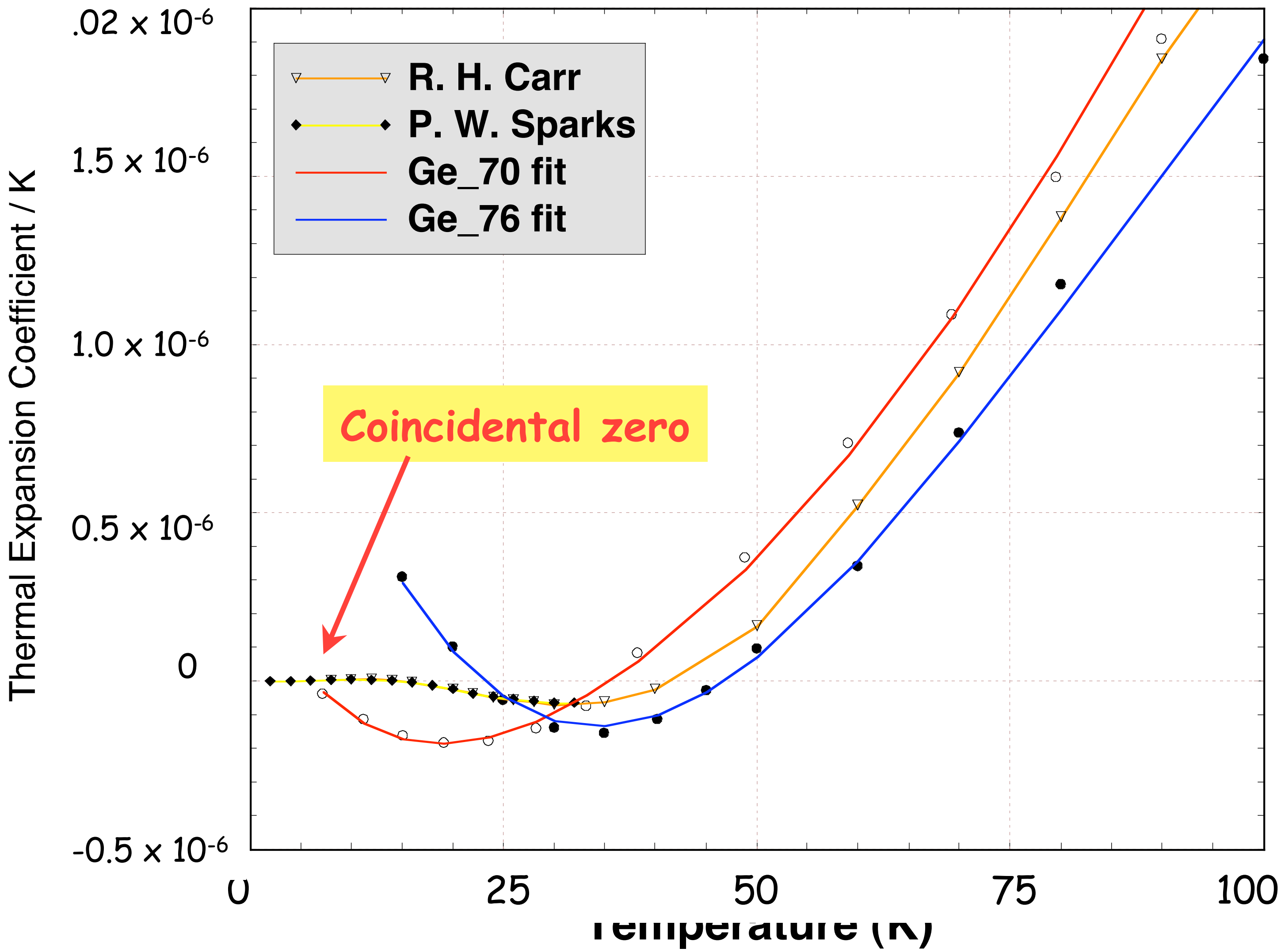

(a-a(70Ge))/a(70Ge)*1e6
10
0
-10
-20
-30
-40
-50
0
50
100
150
200
250
300
Temperature (Celsius)
70
73
74
76


100 < T < 10 K
1
0.5
0
0
1 2
3 4
Energy (eV) + 14323 eV


(a)
Lattice constant (Å)
5.6550
5.6545
5.6540
5.6535
5.6530
5.6525
5.6520
0
50
100
150
200
Temparture (K)
76Ge
70Ge
(b)
Ge_70: NID data (April 2002)
Carr et al, Natural Ge, 1964
Thermal expansion coefficient, □L
4.0 10-6
3.0 10-6
2.0 10-6
1.0 10-6
0.0
-1.0 10-6
0
20
40
60
80
100
120
140
Temperature (K)

Lattice Constant of ^{76}Ge, ^{70}Ge, and Natural Ge

Measured at NID station (except Natural Ge data)



$$a_0 = a \quad + CM^{-1/2}$$

$$U_0(V_0 + \Delta V, M) = U_0(V_0, M) + \frac{1}{2}\left(\Delta V\right)^2 U_0^{''}(V_0, M) + \frac{1}{6}\left(\Delta V\right)^3 U_0^{'''}(V_0, M)$$

$B_0 = V_0 U_0^{''}$ bulk modulus

$$B_0^{'} = -(V_0^2 / B_0) U_0^{'''}$$

$H = \dfrac{P^2}{2M} + 6a_0 B_0 u^2 - 8\sqrt{3} B_0 B_0^{'} u^3 + \ldots$ energy/primitive cell

$$\langle \mathrm{u} \rangle = \frac{h}{2\pi} B_0^{'} (2a_0^3 M B_0)^{-1/2}$$

$$\frac{\Delta a}{a} \frac{1}{\Delta M} = -7.5 \cdot 10^{-6}$$

$$\gamma_i = -\frac{\partial(\ln \omega_i)}{\partial(\ln V)} \quad \text{Gruneisen constant for mode i}$$

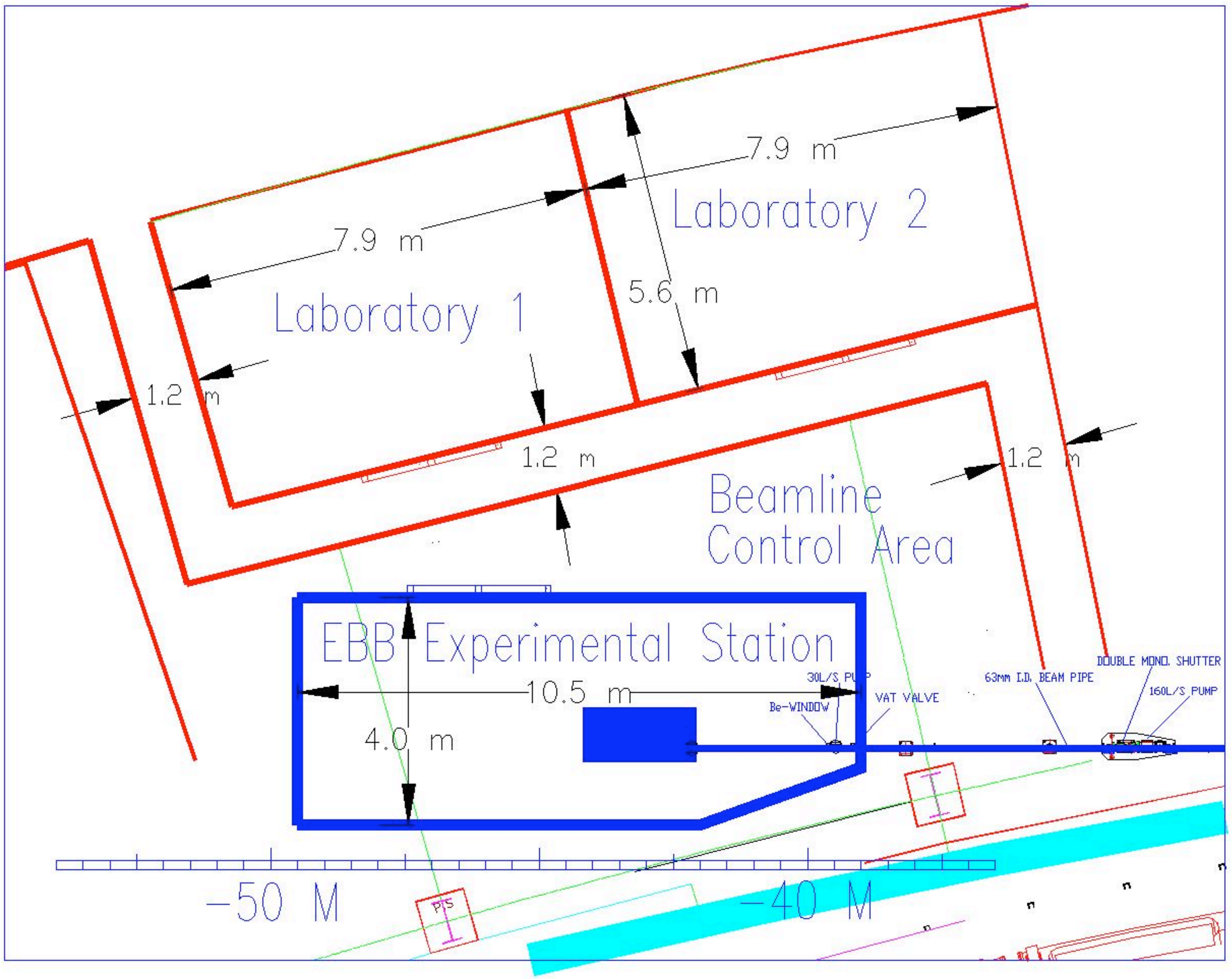
$$\Delta\omega_i = -\gamma_i \omega_i \frac{\Delta V}{V} = -(4\sqrt{3}/a)\gamma_i \omega_i u$$

The thermal expectation value of u to the first order deviation from harmonicity provides a description of temperature dependence of of lattice parameter for 4 different Ge isotopes.

What do we learn ?

- Accurate knowledge of temperature dependence of lattice constant is at the heart of modern density functional theories.
- In order to get it right, all phonon frequencies, and their anomalous behavior has to be predicted.
- Elastic constants are predicted from the slopes of the acoustic modes. So does the bulk modulus.

.. in the near future ….



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